VACUUM

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in
Vacuum Research and Engineering

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THIN AIR

THIN AIR is the element of the vacuum plant manufacturer; his task is to aid the research and production engineer in work, where the production of a rarefied atmosphere is an advantage, by the development of plant systems, leakproof to a high degree, and capable of rapid exhaustion to exceedingly low pressures. As vacuum engineering became more firmly established the work chambers grew larger. Now they have reached the size which permits large scale investigations of the effects of the natural rarefied atmosphere on the performance of aircraft at high altitudes. The low density wind tunnel at Berkeley is a spectacular example. It simulates the atmospheric conditions prevailing many miles up, where pressures are lower than one micron.

The direct high altitude research carried out with the help of rockets, fired from the fields of New Mexico and reaching heights from 120 to 250 miles, also benefits from the progress of vacuum technology. The rockets are fitted with pressure gauges, air sampling devices and other equipment originally developed for, and normally used in, vacuum systems. In addition, there is a host of supplementary techniques and auxiliary instruments, such as air tight sealing of instruments and strain gauges for stress investigations, with which the vacuum engineer has helped to make flying at high altitudes safe.

This is but another example of the indivisibility of science where collaboration between specialists combines to improve our knowledge of the stratosphere and vacuum physics.

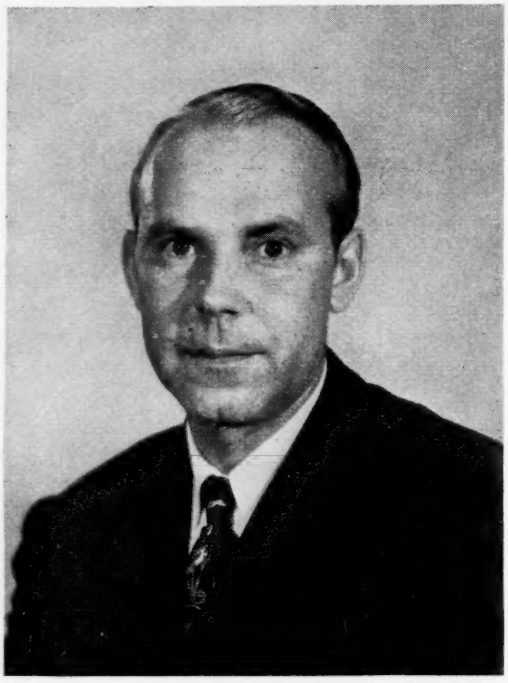
It is to be hoped that the leading part this country has played in aeronautics will continue with the development of low density wind tunnels and the prosecution of fundamental research work which stems from them.

PROTECTIVE OXIDE FILMS

GEORG HASS was the first to use silicon monoxide films for the protection of front-surface aluminium mirrors. Aluminium mirrors protected in this manner had a reflectivity higher than that of rhodium and a comparable mar resistance. This early work of Hass was highly significant, not only for the future of the vacuum evaporation process but also regarding a controversy which has been in progress ever since Winkler reported (in 1890) that silicon monoxide could be obtained in the gaseous state. No doubt Hass' studies of evaporated silicon monoxide films have gone a long way to establish the existence of this compound and the conditions required for its stability.

Lately Hass has turned his attention to the uses of metal oxide films. His most notable and recent achievement in this respect is the preparation of double oxide layers of titanium dioxide and aluminium trioxide on aluminium surfaces obtained simultaneously by anodic treatment. We are very glad indeed to publish an account of this latest work of his in this issue. As in the case of silicon monoxide films the phenomena described in the article may help to clarify the conception, generally held at present in scientific circles, on the mechanism of the oxidation of metals. Modern theory surmises that the growth of an oxide film on a metal surface proceeds with the positively charged metal ions flowing through the oxide film towards the oxide gas surface. Dr. Hass' discovery that an aluminium oxide layer can be oxidized when covered by a titanium dioxide film can hardly be reconciled with that theory.

The results of Dr. Hass' study will be of interest to all who concern themselves with oxidation phenomena, proving once more how closely the problems of the vacuum research worker are interwoven with those of the general physicist and the optical expert.



GEORG HASS

Dr. Rer. Techn. Habil.

Chief, Physics Research,
Radiation Research Laboratory,
Fort Belvoir, Va., U.S.A.

Dr. Hass was born in Hanau (Germany) in 1913. He chose Physics as a profession and began his studies at the Universities of Innsbruck and Freiburg i. Br. (Germany). He was awarded a doctorate in 1937 by the Technical University of Danzig. The following year Dr. Hass established himself as a lecturer of Physics at the same college and remained there in this capacity for a period of five years. His lectures covered a variety of subjects such as corpuscular rays, X-ray and electron diffraction, the physics of metals in general and oxidation and diffusion phenomena of metals in particular. In 1943 he was appointed Assistant Professor of Physics.

For a period Dr. Hass acted as consultant to the research organisations of the Siemens concern and the Telefunken company and as adviser to the Heraeus Platinschmelze and the Vereinigte Aluminium Werke. In 1946 he left Germany and became affiliated to the Engineer Research and Development Laboratories at Fort Belvoir, Va., U.S.A. Five years later this affiliation materialised into his appointment as Chief of the Physics Research of the Radiation Research Laboratory at Fort Belvoir, Va., a position Dr. Hass still holds.

Dr. Hass took up research work as early as 1936. He conducted investigations in the field of light and electron optics but his chief interests are the optical properties and oxidation phenomena of metal surfaces. In the course of time he extended his studies to embrace the structure and behaviour of evaporated thin films (of the single- and multilayer type) and in recent years became widely known for his work on the nature of oxide films.

Dr. Hass is a prolific writer and holds many patents.

preparation, properties and optical applications of THIN FILMS OF TITANIUM DIOXIDE

Georg Hass, Dr. Rer. Techn. Habil.

Engineer Research & Development Laboratories
*Fort Belvoir, Virginia, U.S.A.**

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Summary

THE PAPER presents data on the optical properties, structure and oxidation of evaporated titanium films and discusses the structure, properties, and applications of titanium dioxide coatings prepared by heat oxidation of vacuum deposited titanium and by decomposition of titanium tetrachloride with atmospheric water vapour. It is shown that the rate of deposition and the pressure in the evaporation unit influence greatly the structure and optical properties of evaporated titanium films and of titanium dioxide coatings prepared by heat oxidation of evaporated titanium. Pure titanium films can only be deposited by fast evaporation at good vacuum. Slow evaporation at poor vacuum results in TiO-TiN films with cubic structure. The oxidation of pure titanium films in air at 400° to 450°C results in titanium dioxide films with rutile structure and high n-values while 'poorly deposited titanium films' oxidise at the same temperature to titanium dioxide films with anatase structure and much lower n-values. Rutile is especially suitable as a top film in multilayer combinations. Titanium dioxide films produced by fuming titanium tetrachloride onto hot glass are amorphous if the glass temperature is kept below 280°C and consist of anatase at higher temperatures.

An anodic method for applying reflectance-increasing film pairs of aluminium oxide and titanium dioxide on aluminium mirrors is described.

Sommaire

L'ARTICLE contient les données des propriétés optiques, la structure et l'oxydation de films de titanium évaporées sous vide et l'auteur joint une discussion de la structure, des propriétés et des applications de couches minces de dioxyde de titanium préparées par oxydation à chaud, de titanium déposé par évaporation sous vide et par décomposition de tetrachlorure de titanium par de la vapeur d'eau athmosphérique. La vitesse de déposition et la pression dans la cloche d'évaporation ont une grande influence sur la structure et les propriétés optiques de films de titanium évaporés et de couches en dioxyde de titanium préparées par oxydation à chaud de titanium évaporé. Une évaporation lente ne donne pas des films purs. Elle resulte en un dépôt de TiO-TiN avec structure cubique. L'oxydation de films de titanium dans l'air à 400°C—500°C résultent en des couches minces de dioxyde de titanium sous forme de rutile et index élevé, tandis qu'un film déposé sous des mauvaises conditions oxyde aux mêmes températures sous forme d'anatase et un index beaucoup plus bas. La rutile est surtout convenable comme couche supérieure de films multiples. Les films de dioxyde de titanium produits par fumigation de tetrachlorure de titanium sur le verre chaud sont amorphes, si la température du verre est maintenue en dessous de 280°C et consiste en anatase aux températures plus élevées. Une méthode anodique pour l'application de films doubles augmentants la réflexion, constituées de dioxyde d'aluminium et de dioxyde de titanium sur des mirroirs en aluminium, est décrite.

INTRODUCTION

IN RECENT YEARS, the use of multilayer interference coatings has gained increasing importance in the field of optics. Systems of multiple films have found application in particular for increasing and decreasing the reflectance of metals and dielectrics in various spectral regions, and for making filters with more or less narrow band width in the visible and infra-red. Almost all multilayer combinations consist of dielectric films having alternatively low and high refractive indices. Unfortunately, there are only a few materials suitable for preparing satisfactory non-absorbing dielectric films with sufficiently high refractive index. The materials most frequently used for preparing high index films are zinc sulphide (ZnS)

* M.S. received June, 1953.

and antimony sulphide (Sb_2S_3). These sulphide films, however, even if applied under heat and put through a rather complicated baking process do not have as good mechanical and chemical stability as films of various metal oxides. If mechanical and chemical durability as well as high index of refraction are considered the most desired qualities of a film material, titanium dioxide seems to be most satisfactory.

Titanium dioxide is known to exist in amorphous form, and in three crystalline modifications : Rutile, Anatase, and Brookite. All modifications have a rather high index of refraction in the visible and the useful infra-red region, and are chemically and mechanically very resistant. Unfortunately, however, if titanium dioxide is evaporated in high vacuum, it reduces slightly at the high temperature required for its evaporation and condenses as an absorbing film. A quarter-wavelength thick film of directly evaporated slightly decomposed titanium dioxide reflects up to 30% in the visible and shows less absorption than most 30% reflecting metal coatings. Therefore, films produced by direct evaporation of titanium dioxide have been used as beam-dividers for Fizeau interferometers[1]. For all multilayer applications, however, their light absorption is intolerably high. In recent years, various methods for preparing non-absorbing films of titanium dioxide have been reported :

(1) Vacuum deposition of titanium metal followed by heat oxidation in air at 400°- 450°C*[2].
(2) Fuming of titanium tetrachloride onto heated glass at atmospheric pressure[3,4].
(3) Deposition of titanium dioxide from solutions of various types followed by baking[5,6].
(4) Sputtering of titanium in oxygen[7].
(5) Re-oxidation of slightly reduced titanium dioxide by anodic treatment or heating in air[8].

The refractive indices for titanium dioxide films prepared by the various methods were mostly reported for only one wavelength in the visible and cover a range from n = 2.2 to 2.65. The great variation of the n-values of the films produced under various conditions is mainly due to the fact that the various preparation methods result in the deposition of different modifications of titanium dioxide. Since all of the reported film materials have n-values lower than that of compact rutile ($\bar{n}_D = 2.71$)[9,10] and most values are even lower than that of compact anatase ($\bar{n}_D = 2.53$)[9] it seems certain that the density of all film materials is rather low.

The present paper presents data on the structure and dispersion curves of titanium dioxide films produced by methods (1), (2) and (5) and discusses some new optical applications of these films. The paper also shows that titanium dioxide films with higher n-values and densities than reported previously can be prepared, and that in method (1) the vacuum and evaporation conditions influence greatly the structure and optical properties of the resulting titanium dioxide films.

METHODS USED FOR DETERMINING THE REFRACTIVE INDEX AND THE STRUCTURE OF TITANIUM DIOXIDE FILMS

A spectrophotometric and a polarimetric method were used to determine the refractive indices of titanium dioxide films in the wavelength region from 400 to 2,000 millimicrons.

The spectrophotometric method determines the refractive index of titanium dioxide films on glass from measured wavelength positions and values of reflectance maxima by use of the following relation :

$$n_f = \left[n_g \, (1 + R^{\frac{1}{2}})/(1 - R^{\frac{1}{2}}) \right]^{\frac{1}{2}} \qquad (1)$$

at wavelength of R_{max} position, where n_f = index of the film material, n_g = index of glass or substrate, R = reflectance. To obtain the dispersion curves of the film material a set of coatings ranging in thickness from 300 Å to 2,000 Å was prepared under the same conditions and measured in the wavelength region from 400 to 2,000 millimicrons. Additional values for n_f were calculated from the true film thickness t, measured by multiple-beam interferometry[11], and the optical thickness n t, determined from the position of

* During the war, such films were used in Germany under the name of ' Steinheil 2 '.

the reflectance maxima and minima. For films with less than 2% absorption, and t > 1,000 Å, the n-values of titanium dioxide films determined by both methods agreed within 1%.

The polarimetric method of Abeles[12] was also used for determining the refractive indices of TiO_2 films. In this method the intensity of p-polarised light reflected from a coated glass is compared with that reflected from an uncoated glass surface. At Brewster's angle for the film material φ_o the intensities reflected from the coated and uncoated areas are equal. The refractive index is then given by the simple relation : $n = \tan \varphi_o$. The method is independent of the substrate used and of the film thickness; although greatest accuracy is obtained if the film thickness is in the neighbourhood of a quarter wavelength at Brewster's angle of incidence. If the indices of refraction of the film materials are not very different from that of the substrate, direct observation of a half-coated and half-uncoated surface area through a spectrometer without eyepiece results in n-determination with high accuracy. For the high refracting titanium dioxide, however, a goniphotometric method has to be used to obtain reasonable accuracy. A photocell was attached to the eyepiece of the spectrometer and the reflectance measurements were made from the uncoated and coated glass surface as functions of the angle of incidence. By plotting these values the photometric match point corresponding to Brewster's angle for titanium dioxide could be determined precisely. For homogeneous titanium dioxide films with extremely low light absorption and scattering, the n-values determined by Abeles' method agreed within less than 1% with the indices measured by the spectrophotometric method described above.

The structure of the titanium dioxide films was examined by electron diffraction. The electron diffraction studies were made with the RCA Electron Microscope Type EMU. Thin films of silica, produced by evaporating silicon monoxide, were used as structureless supporting membranes[13]. Thus, the titanium dioxide coatings measured optically and studied by electron diffraction were deposited onto the same glass-like substrates.

TITANIUM DIOXIDE FILMS PREPARED BY HEAT OXIDATION OF EVAPORATED TITANIUM

Extremely hard and durable films of titanium dioxide with refractive indices almost identical with those of pure rutile crystals can be prepared by oxidising evaporated titanium in air at 400° to 450°C. However, the optical and mechanical properties of titanium dioxide films thus prepared depend strongly on the conditions under which titanium is evaporated. Optimum film qualities are obtained only if the titanium evaporation is performed under vacuum and evaporation conditions which result in the deposition of pure and dense titanium. Since titanium getters strongly during its evaporation, pure titanium films can only be deposited by fast evaporation under good vacuum conditions ($p \leq 1 \times 10^{-4}$ mm. Hg). During a slow deposition at poor vacuum ($p \geq 1 \times 10^{-4}$ mm. Hg) titanium is able to getter such a great amount of oxygen and nitrogen that the condensed film material has not the hexagonal structure of titanium but the cubic structure of titanium oxide (TiO) and titanium nitride (TiN). If a pure hexagonal titanium film is oxidised by heating in air at 400° to 450°C, rutile, the hardest and highest refracting modification of titanium dioxide is formed while the oxidation of the cubic TiO or TiO-TiN material in air at the same temperature results in anatase with much lower refractive index.

To prepare a titanium dioxide film with a precisely controlled optical thickness, the following data is required: The reflectance and transmittance of unoxidised titanium as function of thickness, the thickness increase by heat oxidation, and the refractive index of the resulting titanium dioxide coating. In the following parts the preparation and properties of titanium films, their oxidation in air at various temperatures, and their properties and applications after heat oxidation will be discussed in detail.

(1) Preparation of Pure Titanium Films and Their Properties

Tungsten boats made of 0.5 to 1.0 mm. thick sheet material were used to evaporate titanium.

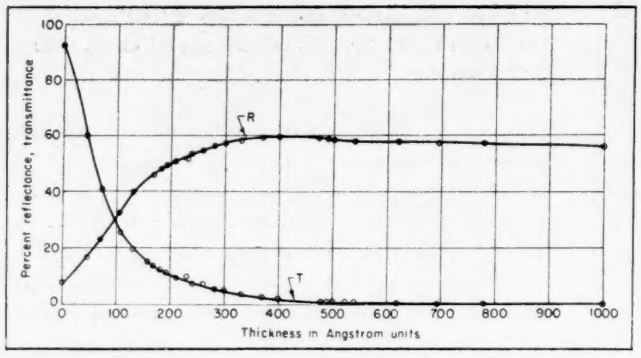

Fig. 1. Reflectance and transmittance of evaporated titanium films as function of thickness at $\lambda = 550$ mμ.

'Commercially pure titanium' with a titanium content exceeding 99.5% was placed in the tungsten boat, melted and completely outgassed before any coating was deposited. The glass plates to be coated were cleaned by a high voltage glow discharge using 5,000 V at 75mA for ten minutes. Then a shield was moved directly below the glass plates and removed after a rather constant evaporation speed was reached and the pressure in the evaporator had decreased to less than 1×10^{-5} mm. Hg. All depositions were carried out at a pressure $p\leqslant1\times10^{-5}$ mm. Hg and with a deposition rate $D\geqslant20$ Å per sec. A great number of titanium coatings ranging in thickness from 100 Å to 1,200 Å were condensed under these conditions on glass and silica and their reflectance and transmittance measured at various wavelengths. Fig. 1 and 2 show the reflectance and transmittance of titanium coatings as functions of film thickness for $\lambda = 550$ millimicrons and $\lambda = 2{,}000$ millimicrons. The film thicknesses were determined with a multiple-beam interferometer and the results shown in the graphs were completely reproducible for films prepared under the conditions mentioned above. The transmittance of titanium is higher at 2,000 millimicrons than it is at 550 millimicrons. A 300 Å thick film transmits 4.5% at 550 millimicrons and 10.5% at 2,000 millimicrons and a film of 1,000 Å, which is almost completely opaque in the visible, still transmits 1.5% at 2,000 millimicrons. Since transmittance measurements during the evaporation are most convenient for controlling the film thicknesses, the preparation of thin titanium films ($t = 100$ Å to 350 Å) should be controlled with a photocell and a 550 millimicron filter, while for the preparation of thicker films ($t = 300$ Å to 1,200 Å) an all-dielectric narrow pass band-filter with peak transmittance at 2,000 millimicrons in combination with a lead sulphide detector is more suitable.

(2) Oxidation of Pure Titanium Films in Air at Various Temperatures

By measuring the thickness of ten titanium films before and after complete heat oxidation the factor of thickness increase r in $t_{TiO_2} = r\, t_{Ti}$ was determined to be $r = 1.78 \pm 0.02$. By assuming normal density for the evaporated titanium, and for the rutile film formed by heat oxidation, r was calculated from :

$$t_{TiO_2} = \frac{M_{TiO_2}}{M_{Ti}} \frac{\rho_{Ti}}{\rho_{TiO_2}} t_{Ti} \qquad (2)$$

to be $r = 1.77$

where M_{TiO_2} the molecular weight of $TiO_2 = 79.9$, M_{Ti}, the atomic weight of Ti = 47.9, ρ_{Ti}, the density of titanium = 4.50 g/cm^3, and ρ_{TiO_2}, the density of rutile = 4.24 g/cm^3.

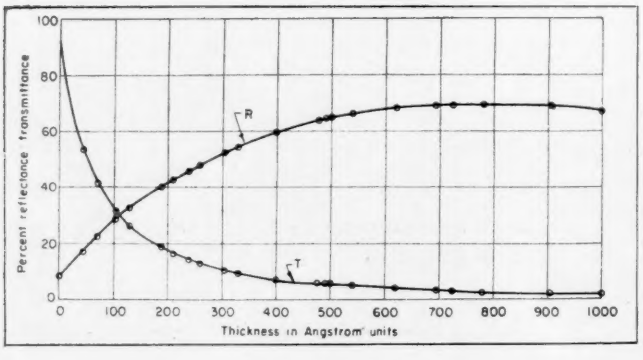

Fig. 2. Reflectance and transmittance of evaporated titanium films as function of thickness at $\lambda = 2{,}000$ mμ.

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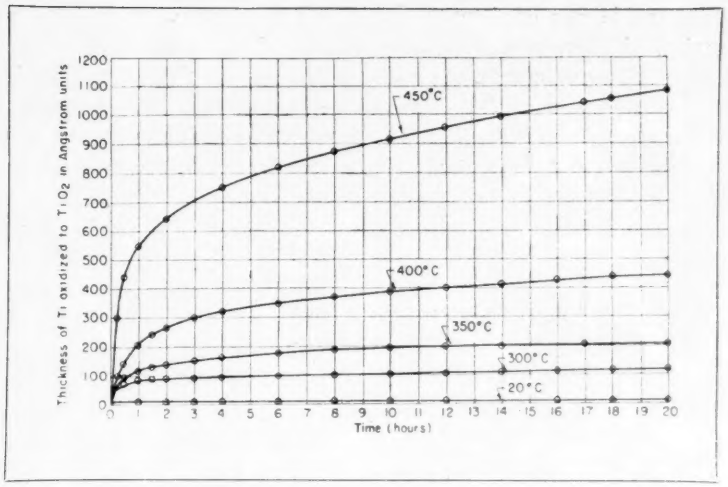

Fig. 3. Oxidation of evaporated titanium films in air at various temperatures.

Since the calculated and directly measured values for the thickness increase by oxidation are in good agreement, and the optical properties of the final rutile films are almost identical with those of compact rutile crystals, it may be assumed that the evaporated titanium coatings have normal density.

Fig. 3 shows the rates of oxidation of evaporated titanium films in air at various temperatures. The measured change in light transmittance and reflectance was used to calculate the amount of titanium converted to titanium dioxide during the oxidation in air. Infra-red light with $\lambda \geqslant 2$ microns was used for the oxidation measurements at higher temperatures. Thus, the thickness of the oxide films formed remained small compared to the wavelength and no undesirable interference effects were introduced. The values for the thickness of titanium oxidised to titanium dioxide have to be multiplied by 1.77 to obtain the thickness of the titanium dioxide films formed on titanium. The oxide film formed on titanium in air at normal temperature is about 20 Å thick after 20 hours exposure. Further growth is very slow and finally ceases almost completely after one month when the oxide film is about 35 Å thick. At room temperature the rate of oxidation of titanium in air is almost equal to that of aluminium. On evapor-

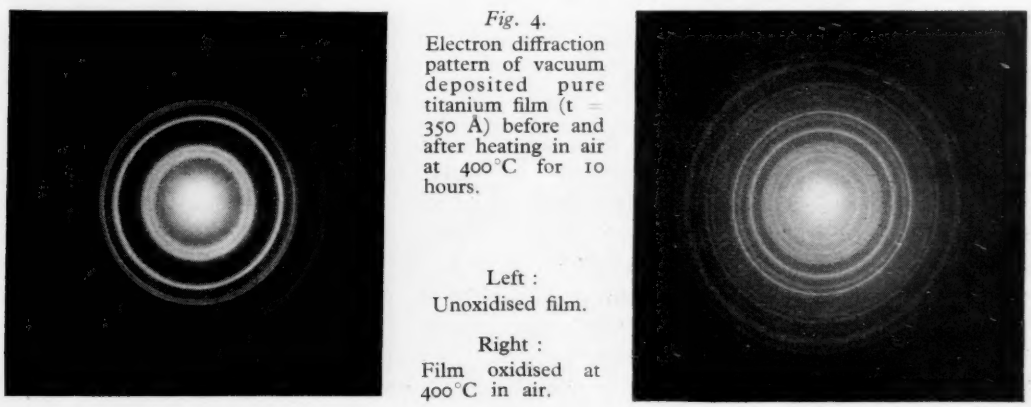

Fig. 4. Electron diffraction pattern of vacuum deposited pure titanium film (t = 350 Å) before and after heating in air at 400°C for 10 hours.

Left : Unoxidised film.

Right : Film oxidised at 400°C in air.

ated aluminium, too, an oxide film of about 15 Å forms in a very short time after first exposure to air and further growth ceases after one month, when a film thickness of 40 Å has been reached[14]. With increasing temperature, however, the oxidation rate of titanium increases much faster than that of aluminium. In Table I the oxidation of titanium in air after two hours at various temperatures is compared with that of aluminium.

Table I. **Thickness of Oxide Films Formed on Evaporated Titanium and Aluminium After Two Hours Exposure to Air at Various Temperatures**

Temp. °C	20	300	350	400	450
t_{TiO_2} in Ångstroems	15	160	250	470	1130
$t_{Al_2O_3}$ in Ångstroems	10	15	20	25	45

The rates of oxidation of evaporated titanium films in air as shown in Fig. 3 are lower than those reported by Gulbransen and Andrew[15] for titanium in dry oxygen. The difference may be explained by the fact that the samples used by Gulbransen and Andrew were abrasively polished and such surfaces are more active toward oxygen and air than the smooth mirror-like surfaces of evaporated films.

The oxidation curves shown in Fig. 3 can be used to estimate time and temperature needed for a complete oxidation of a titanium film with known thickness. Titanium films up to 400 Å in thickness can be oxidised completely by heating at 400°C, while higher temperatures have to be applied for oxidising thicker titanium films to non-absorbing titanium dioxide coatings.

(3) Structure of Evaporated Titanium Films Before and After Heat Oxidation

Fig. 4 shows the electron diffraction pattern of a vacuum deposited, 350 Å thick titanium film before and after oxidation in air at 400°C for ten hours. The pattern of the untreated film is identical with that of pure hexagonal titanium ($a = 2.95$ Å, $c = 4.67$ Å), and the oxidised film shows only rather sharp rings of the tetragonal rutile modification of titanium dioxide ($a = 4.58$ Å, $c = 2.98$ Å). At all temperatures at which a measurable oxide film was formed on pure titanium (temp. > 200°C), the film structure was that of the rutile. Rutile once formed could not be changed to any other modification of titanium dioxide at any temperature. These results are in agreement with the report of Hickman and Gulbransen[16]. They also found titanium dioxide only in the form of rutile on titanium heated in oxygen from 300°C to 700°C. Furthermore, the results support the interpretation that rutile is the only stable modification of titanium dioxide and that the other modifications, brookite and the very frequently appearing anatase, are unstable transition forms. Some of the circumstances under which anatase is formed and the conditions under which anatase is converted to rutile will be discussed in the later part of this paper.

(4) Optical Properties of Titanium Dioxide Films Produced by Heat Oxidation of Pure Titanium Films.

Titanium dioxide coatings prepared by complete oxidation of pure titanium films in air at 400°C to 450°C are optically very smooth and uniform, and show at quarter-wavelength thickness less than 0.5% absorption in the measured wavelength region from 400 to 2,000 millimicrons. Fig. 5 shows the refractive index of such films as function of wavelength. As indicated on the graph, all three methods mentioned above were used to determine the dispersion curve. Values for the mean refractive indices of a single crystal of rutile $\bar{n} = (2n_o + n_e)/3$, are plotted on the same graph. The refractive indices of the film material average about 1 to 1.5% lower than those of a rutile single crystal.

Fig. 6 shows the maximum reflectance R_{max} obtained with quarter-wavelength thick films of titanium dioxide on glass with $n_g = 1.51$ as function of wavelength from 400 to 800 millimicrons. In this region R_{max} decreases with increasing wavelength from 52.9% to 41.4%. The reflectance and transmittance of a titanium dioxide film having its quarter-wavelength position $\lambda = 550$ millimicrons are shown for the same wavelength region. All reflectance values in Fig. 6 are directly measured and not corrected for back reflection.

To prepare a quarter-wavelength thick film of

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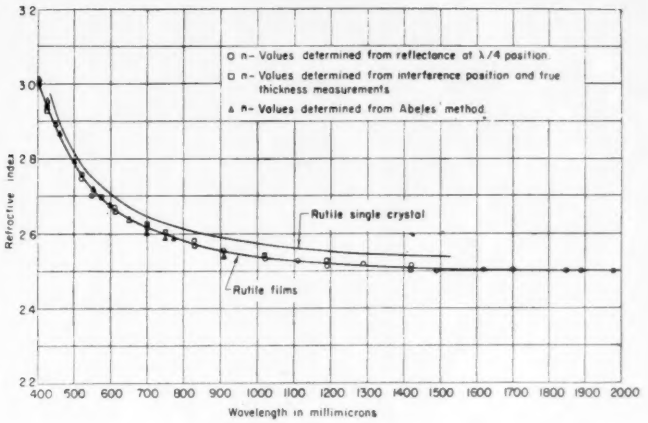

○ n- Values determined from reflectance at λ/4 position.
□ n- Values determined from interference position and true thickness measurements
△ n- Values determined from Abeles' method.
Rutile single crystal
Rutile films
Refractive index
Wavelength in millimicrons

Fig. 5. Refractive index of rutile films and of a rutile single crystal as function of wavelength from 400 mμ to 2,000 mμ.

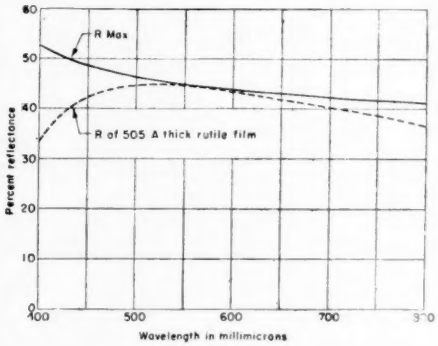


Fig. 6. Maximum reflectance obtained with $\lambda/4$ thick film of rutile on glass with (n_g = 1.51) as function of wavelength from 400 to 800 mμ, and reflectance curve of a rutile film with $\lambda/4$ position at λ = 550 mμ.

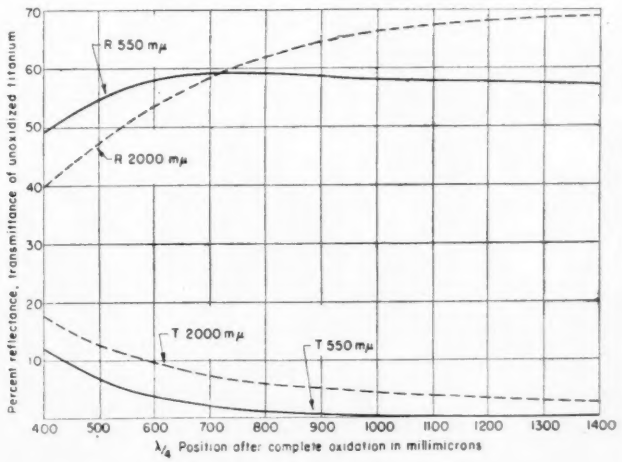

R 550 mμ
R 2000 mμ
T 2000 mμ
T 550 mμ
Percent reflectance, transmittance of unoxidized titanium
λ/4 Position after complete oxidation in millimicrons

Fig. 7. Reflectance and transmittance of unoxidised titanium at λ = 550 mμ and λ = 2,000 mμ as function of $\lambda/4$ position of resulting rutile film.

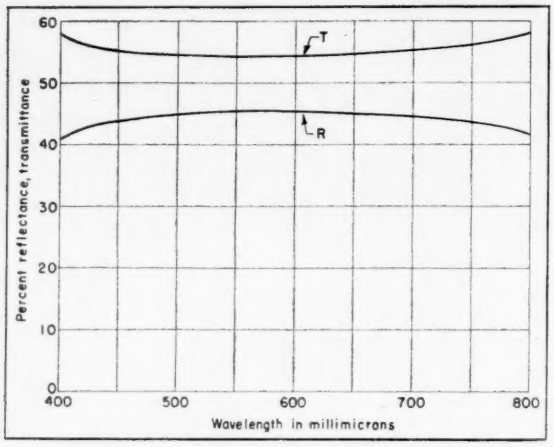
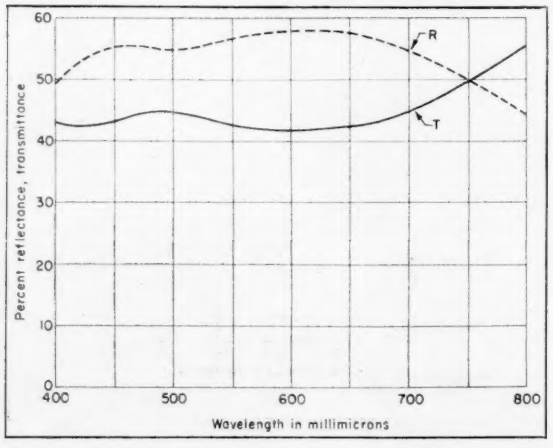


Fig. 8. Reflectance and transmittance of glass (n_g = 1.51) coated with MgF_2 (nt = $\lambda/2$ at λ = 450 mμ) and rutile (nt = $\lambda/4$ at λ = 530 mμ) as function of wavelength from 400 to 800 mμ.

titanium dioxide for λ = 550 millimicrons, titanium is evaporated onto glass until its transmittance at λ = 550 millimicrons has decreased to 5.0%. A film with such transmittance has a thickness of 285 Å (Fig. 1) and oxidises completely when heated in air at 400°C to 420°C for a few hours. After oxidation the film thickness is increased by a factor of 1.77

Fig. 9. Reflectance and transmittance of glass (n_g = 1.51) coated with a triple film made up of $\lambda/2$ of SiO, $\lambda/4$ of MgF2 and $\lambda/4$ of rutile (at λ = 500 mμ) as function of wavelength from 400 to 800 mμ.



resulting in a titanium dioxide film of 505 Å. Its optical thickness n t is therefore equal to 1,370 Å or to a quarter-wavelength of light with λ = 550 millimicrons. Fig. 7 presents all information needed for a controlled preparation of quarter-wavelength thick films of titanium dioxide at different wavelengths. The reflectance and transmittance of unoxidised evaporated titanium at λ = 550 millimicrons and λ = 2000 millimicrons are plotted against the quarter-wavelength position of the resulting titanium dioxide films.

(5) Optical Applications of Titanium Dioxide Films Prepared by Heat Oxidation of Pure Evaporated Titanium

The excellent mechanical, chemical and optical properties of titanium dioxide prepared by heat oxidation of evaporated titanium justify its applications as optical coatings, in spite of the fact that its preparation requires an extra operation after the evaporation cycle. In addition to its use as a single-layer beam-splitter coating, titanium dioxide is especially suitable as a top film in multilayer combinations, since this application requires only one heat treatment after the evaporation process. A great variety of extremely durable beam-divider coatings can be prepared by using titanium dioxide in combination with magnesium fluoride (n_D = 1.38) and silicon monoxide (n_D = 1.97).* With quarter wavelength thick films of these materials the following reflectance and transmittance values can be obtained on glass with n_g = 1.51 at λ = 550 millimicrons :

Film Materials on Glass	*R%*	*T%*
TiO_2	44.5	55.0
MgF_2 —TiO_2	50.0	49.5
SiO —MgF — TiO_2	61.0	38.0
MgF_2 —SiO — MgF_2 — TiO_2	66.0	33.0
SiO —MgF_2 — SiO —MgF_2—TiO_2	74.0	24.0

* The refractive indices of ' SiO films ' depend strongly on the evaporation conditions. True SiO with n_D = 1.97 is obtained by fast deposition (10-15 Å per sec.) at p ~ 10^{-5}mm. Hg. Slower deposition at about 1×10^{-4}mm. Hg of air or oxygen results in films with much lower n-values[17].

Fig. 8 and 9 show the reflection and transmission characteristics of two film combinations on glass which are substantially achromatic throughout the visible spectrum. The first combination consists of a film of magnesium fluoride having a thickness of half a wavelength at $\lambda = 450$ millimicrons, and a film of titanium dioxide one quarter-wavelength thick at $\lambda = 530$ millimicrons. This two-layer coating has a reflectance of more than 44% without appreciable loss of light over a rather extended spectral region.*

The second combination is designed for higher reflectance and consists of three layers. The first one on glass is silicon monoxide, having a thickness of half a wavelength and the other two are magnesium fluoride and titanium dioxide with $n\,t = \lambda/4$ at $\lambda = 500$ millimicrons. This three-layer combination has a reflectance of more than 50% throughout the visible spectrum. All multilayer combinations described above are especially durable if the glass to be coated is heated during the deposition of the films. A temperature of 250° to 300°C, which is most frequently used for producing hard, low reflecting coatings of magnesium fluoride on glass gives best results.

For using more than one titanium dioxide film in a multilayer combination it is practical to oxidise the evaporated titanium directly in the evaporation chamber by heat treatment at a few microns of oxygen. At this low pressure titanium oxidises almost as rapidly as it does in air at normal pressure. The method has the advantage that evaporation and oxidation of titanium can be carried out without breaking the vacuum. The following procedure was used to prepare multilayer coatings of alternate films of titanium dioxide and magnesium fluoride : The glass to be coated was heated to about 300°C and titanium of the desired thickness was deposited. The coated glass was then heated to more than 400°C and exposed to oxygen of a few microns pressure which was introduced through a needle valve. After the titanium was completely oxidised (about 30 minutes at 440°C for a $\lambda/4$ film at 550 millimicrons) the temperature was lowered to 300°C, the pressure decreased and magnesium fluoride and a second layer of titanium deposited. Then the oxidation of titanium was again performed and new layers of magnesium fluoride and titanium added and treated as described. With five alternate $\lambda/4$ thick layers of titanium dioxide and magnesium fluoride on glass 93.5 per cent. reflectance with only 1 per cent. absorption was obtained. This high reflecting and low absorbing multilayer coating is especially suitable as first surface in reflection interferometry. An optical flat coated with the described multilayer combination has been used to measure film thicknesses for more than a year without damage. It produces extremely sharp wedge fringes with high contrast due to its low absorption.

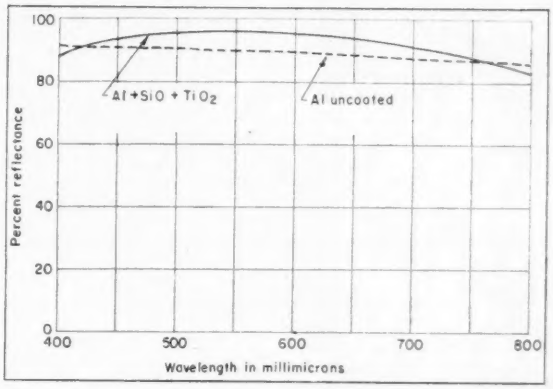


Fig. 10. Reflectance of evaporated aluminium with and without reflectance-increasing film pair of silicon monoxide and rutile as function of wavelength from 400 to 800 mμ.

The reflectance of metals can also be increased by the application of pairs of interference films with alternatively low and high refractive indices. Each film must be effectively one quarter-wavelength thick to obtain highest reflectance (due to the phase change at the metal surface an effective $\lambda/4$ thick film on aluminium is thinner than a true $\lambda/4$ thick one). Fig. 10 shows the visible reflectance of an evaporated aluminium mirror coated with a very durable,

* The use of a half-wave film to achromatise the reflectance of a film combination of quarter wave films was first described by A. F. Turner in a paper presented to the Detroit Section of the Optical Society of America, January 22nd, 1951.

reflectance increasing film pair of silicon monoxide and titanium dioxide. Silicon monoxide was evaporated slowly at 1×10^{-4} mm. Hg of oxygen to produce a film material with rather low refractive index. Titanium was condensed rapidly at very low pressure and converted to titanium dioxide by heating in air at 400°C. The underlying aluminium coating remains uneffected by the heat treatment necessary for oxidising the titanium. Coatings of silicon monoxide and titanium dioxide were applied in the same way to highly polished surfaces of stellite (hard alloy of Co, Cr, and W). They increased the reflectance by more than 20% and prevented surface corrosion up to 600°C.

(6) Structure and Properties of 'Poorly Deposited Titanium' Films Before and After Heat Oxidation

If titanium is evaporated slowly under poor vacuum conditions ($p \geqslant 1 \times 10^{-4}$ mm. Hg) it getters great amounts of gases, especially oxygen and nitrogen, and the deposited films consist mostly of solid solution of titanium with oxygen and nitrogen. Such films have a different structure and are optically more transparent than pure titanium films. Fig. 11 shows the electron diffraction pattern of 'poorly deposited titanium' before and after oxidation in air at 400° to 450°C, and Table II compares the interplanar distances (d-values from Bragg's equation) calculated from the diffraction pattern of a pure titanium film (Fig. 4) with that of a 'poorly deposited' one. Both films were about 350 Å thick. The first film was deposited in 15 seconds at $p \leqslant 1 \times 10^{-5}$mm. Hg and had a transmittance of 1.5% at 550 millimicrons. The second film was condensed in three minutes at about 1×10^{-4}mm. Hg of air and had a light transmission of about 11%. The d-values of the first film are identical with those of pure

Table II. **Interplanar Distances Calculated from the Diffraction Patterns of Pure Titanium and 'Poorly Deposited Titanium' Films**

Ti 350 Å *Thick Deposited in* 15 *sec. at* $p < 1 \times 10^{-5}$*mm. Hg of Air*		*Ti* 350 Å *Thick Deposited in* 3 *min at* 1×10^{-4}*mm. Hg of Air*	
d(Å)	I	d(Å)	I
2.55	str	2.43	str
2 34	str.	2.10	v.str.
2.24	v.str.	1.48	v.str.
1.72	m.	1.27	m.
1.48	str.	1.21	m.
1.34	m.	1.05	w.
1.26	m.	0.965	m.
1.23	m.	0.940	str.
1.18	v.w.	0.860	m.
1.13	v.w.	0.810	m.
1.07	v.w.		
0.99	w.		
0.95	m.		
0.92	m.		

d(A), interplanar distances in Angstroem units; I, estimated intensities: very strong (v.str.), strong (str.), medium (m), weak (w), and very weak (v.w.).

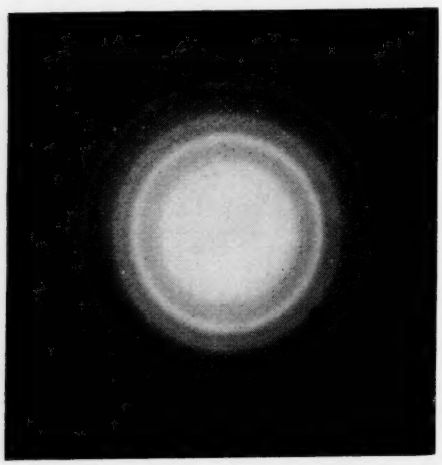
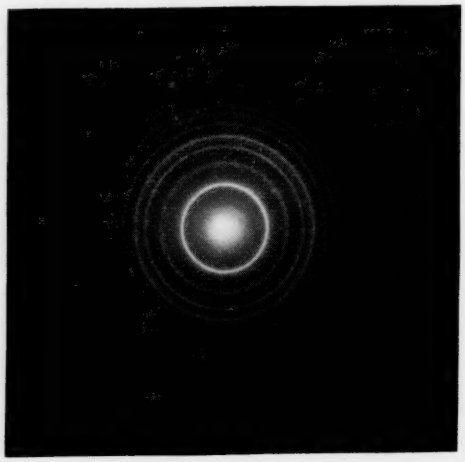


Fig. 11. Electron diffraction pattern of 'poorly deposited titanium' (350 Å in 3 min. at 1×10^{-4}mm. Hg of air) before and after heating in air at 400° at 450°C for 10 hours.

Left : Untreated film.

Right : Film heated in air at 400°C.



hexagonal titanium, while the d-values of the second film are those of a NaCl-type cubic material with a = 4.20 Å. Since TiN and TiO are both cubic with a = 4.23 Å[18] and 4.166 Å[19] respectively, the pattern of the 'poorly deposited' film is identical with that of a solid solution of TiN and TiO.

Films with the structure and lattice constant of TiO could be obtained by evaporating titanium slowly at about 1×10^{-4} mm. Hg of oxygen. By heat treatment in air at 400° to 450°C the 'poorly deposited films' can also be converted to non-absorbing titanium dioxide. However, the resulting films have much lower refractive indices than those prepared from pure titanium and their diffraction pattern shows only rings of the anatase modification of titanium dioxide.

Fig. 12 shows the dispersion curve of such a completely non-absorbing anatase film as determined by Abeles' method. The n-values are even much lower than those of an anatase single crystal. This can only be explained by a rather low density of the film material. By heating the anatase film (Fig. 12) on SiO_2 substrate for two hours at 600°C its refractive index at λ = 580 millimicrons increased from 2.37 to 2.50 and its diffraction pattern showed rings of anatase and rutile with almost equal intensities. After further heating for two hours at 800°C all anatase was converted to rutile and the refractive index increased to 2.65.

TITANIUM DIOXIDE FILMS PREPARED BY FUMING TITANIUM TETRACHLORIDE ON TO HEATED GLASS AT ATMOSPHERIC PRESSURE

The method of depositing titanium dioxide films by decomposing titanium tetrachloride with atmospheric water vapour has been described recently by Geffcken[4] and Banning[20]. A dense white cloud of titanium dioxide forms when a stream of normal air which contains enough water vapour is passed over the surface of heated titanium tetrachloride or is mixed with vapour of titanium tetrachloride. If a glass plate is placed in this cloud, titanium dioxide condenses upon it and forms a film. The properties and structure of the films deposited in this way depend strongly on the temperature of the glass. A soft and loose film is formed on unheated glass. The hardness, density and refractive index of the film material increases with increasing temperature of the glass plate. Banning[20] obtained best results by heating the glass plate to 200°C. To determine the effect of the glass temperature on the optical properties and structure of titanium dioxide films prepared by the gas reaction process, a great number of coatings were condensed on glass, silica substrates and rock salt of various temperatures and studied optically and with electron diffraction. All films condensed on substrates at temperatures below 280°C were amorphous, and all titanium dioxide deposited at higher temperatures had the structure of anatase. Fig. 13 shows the electron diffraction patterns of titanium dioxide films prepared by fuming titanium tetrachloride onto silica substrates heated to 200°C and 300°C. The pattern of the film condensed at 200°C consists of several halos while the film condensed at 300°C shows sharp rings of the anatase modification of titanium dioxide. The pattern of the '300°C film' is identical with that of heat oxidised 'poorly evaporated titanium' (Fig. 11). The amorphous film also changed to anatase when heated to 300°C or more. Films deposited on glass, silica and rocksalt had the same structure. A long heat treatment at 900°C was needed to convert anatase of the gas reaction process to rutile, while anatase produced by oxidising 'poorly evaporated titanium

Fig. 12. Dispersion curve of an anatase film prepared by heat oxidation of 'poorly deposited titanium' at 400°C to 450°C (Ti evaporated in 3 min. at 1×10^{-4} mm. Hg of air to t = 350 Å).

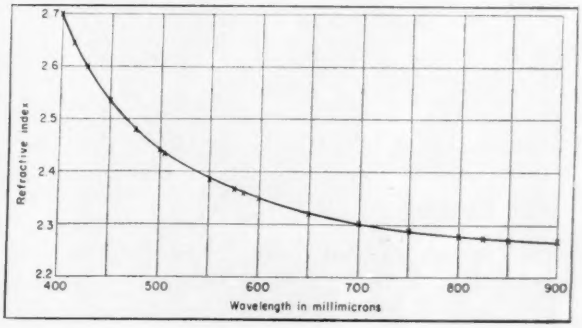
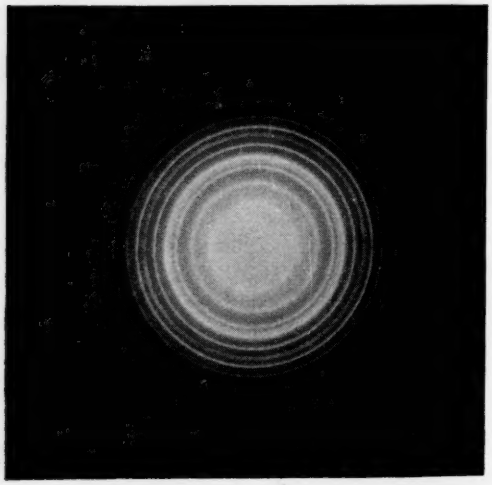


Fig. 13. Above : Substrate at 200°C, film amorphous.

Electron diffraction patterns of titanium dioxide films prepared by fuming titanium tetrachloride on to SiO_2 substrates heated to 200°C and 300°C.

Below : Substrate 300°C, film anatase.



films ' at 400° to 450°C started to change to rutile at about 600°C.

The refractive indices of titanium dioxide films prepared from titanium tetrachloride are rather low. In Table III the average n-values of amorphous coatings condensed at 200°C and of anatase films deposited at 300°C are compared with those of an anatase single crystal at five different wavelengths.

Table III. **Refractive Indices of TiO_2 Films Prepared by Fuming $TiCl_4$ onto Glass at Various Temperatures and of an Anatase Single Crystal**

Material	*n at* 450mμ	*n at* 500mμ	*n at* 550mμ	*n at* 600mμ	*n at* 700mμ
TiO_2 deposited at 200°C	2.40	2.34	2.30	2.28	2.25
TiO_2 deposited at 300°C	2.45	2.39	2.34	2.32	2.29
Anatase single crys.	2.703	2.615	2.565	2.532	2.485

Titanium dioxide films prepared by the gas reaction process are rougher and mechanically less durable than the much denser rutile coatings produced by heat oxidation of evaporated titanium.

ANODIC METHOD FOR PREPARING REFLECTANCE INCREASING FILM PAIRS OF TITANIUM DIOXIDE AND ALUMINIUM OXIDE ON EVAPORATED ALUMINIUM MIRRORS*

The direct evaporation of titanium dioxide results in the deposition of a slightly reduced, strongly absorbing film material. The amount of decomposition and light absorption in the deposited film depends on the evaporation conditions. It increases with the speed of evaporation or the temperature of evaporation source, and decreases with the amount of air present during the deposition. The absorbing, slightly reduced titanium dioxide (TiO_x) can be re-oxidised to non-absorbing TiO_2 by heating in air and

* A paper on this process was presented by A. P. Bradford and G. Hass at the meeting of the Optical Society of America in New York, Y.N., March 19-21, 1953, and will be published in the *Journal of Optical Society of America*.

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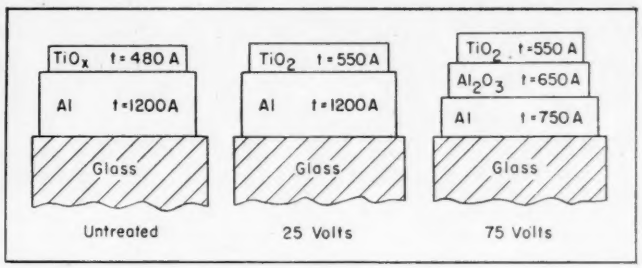

Fig. 15. Layer arrangement on mirror form coated with Al and partially decomposed TiO_2 before and after anodic oxidation at 25 volts and 75 volts to illustrate preparation of Al-Al_2O_3-TiO_2 mirrors with highest visible reflectance.

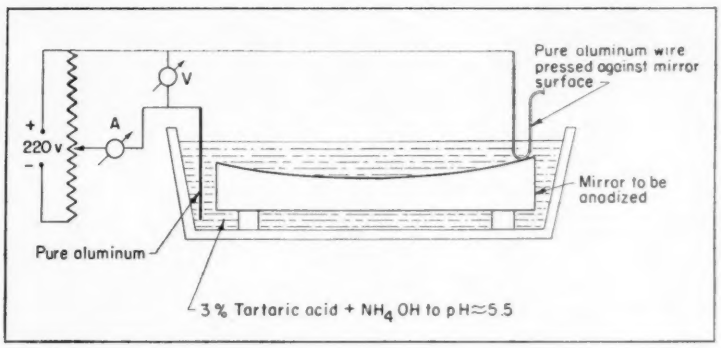

Fig. 14. Sketch of arrangement used for anodic preparation of Al_2O_3-TiO_2 film-pairs on evaporated aluminium.

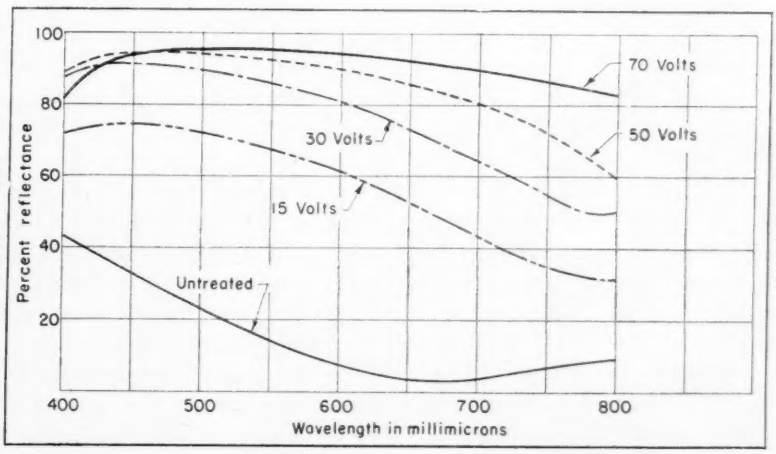

Fig. 16. Reflectance of Al coated with evaporated, partially decomposed TiO_2, 480 Å thick before and after anodic oxidation with 15 volts, 30 volts, 50 volts and 70 volts as function of wavelength from 400 mμ to 800 mμ.

by anodic oxidation. The anodic process is especially suitable for preparing extremely durable reflectance-increasing film pairs of aluminium oxide (Al_2O_3) and TiO_2 on evaporated aluminium mirrors. If TiO_x is condensed on an aluminium mirror the aluminium reflectance decreases to $R_{min} \leqslant 10\%$ due to the high absorption and high index refraction of the deposited TiO_x film. By anodic treatment the absorbing TiO_x on aluminium can be re-oxidised to non-absorbing TiO_2 and part of the aluminium can be converted to Al_2O_3 underneath the TiO_2. This results in a reflectance-increasing low index-high index film pair.

Fig. 14 shows a sketch of the arrangement used for the anodic treatment. The anodic method here is identical with the one used for preparing structureless supporting membranes of aluminium oxide for electron microscopy[21], and for applying hard oxide films on aluminium mirrors[22]. Aluminium coated with TiO_x was made the anode in an electrolyte containing oxygen-bearing anions. Ammonium tartrate, in which neither TiO_2 nor Al_2O_3 are soluble was used as an electrolyte. When voltage is applied, TiO_2 and Al_2O_3 form anodic barrier layers on the aluminium surface so that the high current which flows initially decreases to less than 20μA per cm^2 in 1-2 minutes, indicating that the oxidation has practically ceased. For each film material, the thickness of the oxide film formed depends only on, and increases linearly with, the applied voltage. The rate of TiO_2 formation was determined to be 22 Å per Volt while Al_2O_3, which starts forming after all of the TiO_x is oxidised to TiO_2, increased by 13 Å per Volt. For treating a high-grade mirror, the mirror form was placed completely into the electrolyte and pure aluminium wires were pressed against its surface to make contact. By using this method no contact marks could be seen after the oxidation was performed.

Fig. 15 illustrates the preparation of Al_2O_3-TiO_2 protected aluminium mirrors with highest visible reflectance. It shows the layer arrangement on a mirror form before and after anodic treatments. Aluminium was condensed for double the time it took to deposit an opaque film. This resulted in about 1,200 Å of aluminium. Then TiO_2 was evaporated until the aluminium reflectance reached a minimum value at $\lambda = 625$ millimicrons. This resulted in a strongly absorbing TiO_x film of about 480 Å. By anodising with 25 Volts all TiO_x oxidised to TiO_2 and the film thickness increased to 550 Å. By increasing the voltage to 75, 650 Å of Al_2O_3 were formed underneath the TiO_2. In practice, for producing high reflecting mirrors, 75 Volts were applied directly for about two minutes. Fig. 16 shows the reflectance of aluminium coated with about 480 Å of TiO_x before and after anodic treatments at various voltages as function of wavelength from 400 to 800 millimicrons. TiO_x was deposited until the aluminium reflectance reached a minimum of about 2% at $\lambda = 625$ millimicrons. When air was admitted, part of the TiO_x oxidised and the wavelength position of R_{min} shifted to longer wavelength. After applying 15 Volts, more than half of the TiO_x was oxidised, and the light absorption was strongly decreased. With 30 Volts all absorption had disappeared, and a reflectance maximum of more than 90% was obtained at $\lambda = 450$ millimicrons. With further increase of the voltage the reflectance maximum increased and shifted to longer wavelength and with 70 Volts the reflectance of a great part of the visible spectrum was higher than 95%.

To prepare mirrors with highest reflectance in the near infra-red at $\lambda = 1.0$ to 1.2 microns, TiO_2 was evaporated onto rather thick aluminium until its reflectance reached the first maximum at $\lambda = 600$ millimicrons and anodised with 160 Volts. Thus, an Al_2O_3 film effectively one quarter-wavelength thick at $\lambda = 1.1$ microns was formed underneath a quarter-wave thick film of TiO_2, resulting in a near infra-red reflectance of 98%.

The abrasion and corrosion resistance of Al_2O_3–TiO_2 protected aluminium is good. The films are not damaged by strong rubbing with linen, and can be boiled in 5% NaCl solution for a period of one hour without change in reflectance.

The electron diffraction patterns of TiO_x and anodically produced TiO_2 films showed only very diffuse rings, characteristic for amorphous materials. The amorphous films changed to anatase when heated in air at 450° to 500°C.

The refractive indices of the anodically produced

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TiO_2 films are slightly higher than those of TiO_2 films prepared by decomposing titanium tetrachloride, and are almost identical to those of the anatase film shown in Fig. 12.

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THE DISTRIBUTION OF THIN FILMS CONDENSED ON SURFACES by the Vacuum Evaporation Method

L. Holland & W. Steckelmacher, B.Sc.

*Research Laboratories, W. Edwards & Co. (London) Ltd. Manor Royal, Crawley, Sussex**

Summary

THE PROBLEM of determining the laws of distribution of thin film deposits evaporated in vacuum is discussed in detail. From various types of practical sources, idealised sources are evolved for which the emission of vapour can be calculated. As a fundamental law, the 'cosine law' of Knudsen is recognised. The calculation is extended to point sources, long strip sources, wire sources and ring sources. Evaporation on to plane and spherical receiving surfaces is considered. The practical application of these results to the evaporation of zinc sulphide and magnesium fluoride from strip heaters, and aluminium from spiral filaments is considered together with experimental confirmation. The range of validity of the results is discussed.

Sommaire

DISCUSSION DETAILLES du problème de la détermination des lois regissant le dépôt en film mince evaporé sous vide. Partant de differents types existants des souces d'évaporation idéales sont derivées, par lesquelles l'emission de vapeur peut être calculée. Une loi fondamentale, la loi du consinus de Knudsen est reconnue. Les calculs sont étendus aux sources ponctuelles, élonguées, filiformes et annullaires. L'évaporation sur des surfaces sphériques et planes est considérée. Les applications pratiques de ces resultats sont pris en consideration pour l'évaporation de fulfure de zinc ou de fluorure de magnesium de sources en ruban et d'aluminium au départ de filaments en spirale. Des éxperiences ont été exécutées et une critique des résultats est ajoutée.

INTRODUCTION

TO OBTAIN A UNIFORM deposit on a large surface one has to consider the shape of the surface and the arrangement of the evaporation source or sources relative to the surface. In addition to these obvious considerations it is also important to take into account the mode of evaporation from the source, namely, whether evaporation takes place uniformly in all directions or if there are any preferred directions.

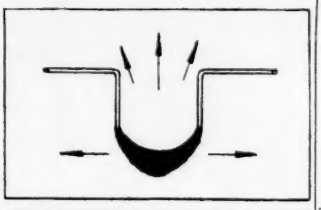
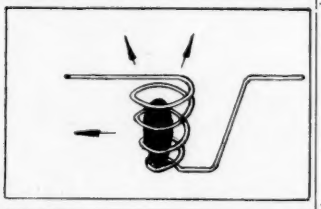
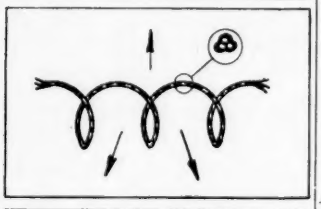
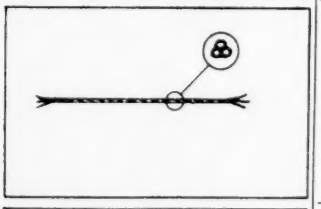
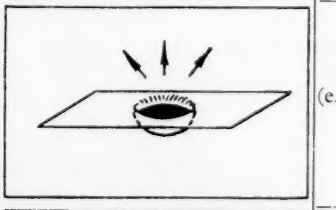
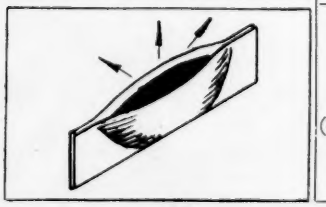
Let us look at some of the many types of sources which are in current use. They are shown diagrammatically in Fig. 1. It is clear that the choice of any particular source arrangement is usually dependent on a number of factors connected with the material to be evaporated, the material holding the evaporant, and the method of heating employed. Thus the material to be evaporated may take the form of granules, powder, wire or sheet. The temperature at which the material has sufficient vapour pressure to evaporate may cover a very wide range and the interaction of the materials to be evaporated with the evaporant support may be a deciding factor. Also, some materials have alloying and wetting properties while others tend to form globules. A few typical effects are described in Fig. 1.

In general we shall assume that evaporation takes place according to Knudsen's cosine law[1,2]. This is the gas-kinetic analogy to Lambert's law for radiation. Thus, some of the theoretical results deduced below have their analogy in illumination engineering. Since the introduction of strip lighting this subject has been extended beyond consideration of point sources [9-11].

We will now consider the assumptions to be made

* M.S. Received August, 1953.

Fig. 1. Typical Evaporation Sources in Current Use with Examples of Their Application.

Type of Filaments	*Designation and Description*	*Typical Applications*	*Materials used as Evaporants*	*Remarks*
	(a) Hairpin (e.g.W-Wire)	Shadow Casting for Electron Microscopy	Gold and Platinum-Palladium Alloy	
	(b) Wire Basket (e.g.W-Wire)	Evaporation of Large Granules of Materials	Chromium	Sublimation or wetting depends on rate of heating and presence of oxide.
	(c) Spiral of Single or Stranded Wire (e.g.W-Wire)	Evaporation of materials which wet easily or have been fixed to the filament by Electrodeposition	Aluminium and Rhodium	Aluminium wets alloys. Rhodium is fixed by plating.
	(d) Straight Stranded Wire (e.g.W-Wire)	Evaporation of materials which wet easily or have been fixed to the filament by Electrodeposition	Aluminium and Rhodium	Aluminium wets alloys. Rhodium is fixed by plating.
	(e) Dimpled Sheet (e.g.Mo or Ta Sheet)	Evaporation of Small Granules and Fine Powder	Silver Copper Magnesium-Fluoride Zinc-Sulphide	Silver forms globules. Copper wets only at high temperatures. Magnesium-Fluoride wets in the molten state. Zinc-Sulphide sublimes.
	(f) Trough made of Folded Sheet (e.g.Mo or Ta Sheet)	Evaporation of Small Granules and Fine Powder	Magnesium-Fluoride Zinc-Sulphide	Magnesium-Fluoride wets in the molten state. Zinc-Sulphide sublimes.

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about the sources in more detail. It will be convenient at this stage to classify evaporation sources as follows :—

(i) *Point Sources.* Consider an emitting sphere, small in comparison with the distance to the receiving surface. It is assumed to have a uniform surface temperature. From the symmetry of this arrangement it follows that evaporation would take place uniformly in all directions.

It is clear that a source described in these terms would be extremely difficult to realise, but all we are concerned with here, is that evaporation is to take place uniformly in all directions, and this is approximately fulfilled by the practical sources (a), (b) and (c) in Fig. 1.

(ii) *Directed Surface Sources.* Consider a small plane emitting surface or sheet of uniform temperature. Evaporation is assumed to take place uniformly according to Knudsen's assumptions for evaporation from such surfaces at low pressures. For details the reader is referred to books on the kinetic theory of gases [3-5]. This law of evaporation involves, for any direction under investigation, the cosine of the emission angle so that the surface has directional properties. In practice, sources of type (e) and (f) may approximate to this.

(iii) *Wire Sources.* Consider the source in the form of a small emitting cylinder having a diameter small compared with other distances. This is again assumed to have a uniform surface temperature. This type of source must behave in some respects intermediate between (i) and (ii), but it would be still expected to have some directional properties. A practical example is shown in (d) in Fig. 1.

We shall also make two general assumptions about the evaporation rate :

(1) It is maintained uniform throughout deposition.

(2) It is sufficiently low so that the effect of collisions of vapour atoms in the vicinity of the source can be neglected.

The second assumption is one of the necessary conditions for Knudsen's cosine law to be applicable.

It is clear that from the point of view of using the minimum number of assumptions about sources, one need only postulate sources of the directed surface type, *i.e.* type (ii) as type (i) and (iii) can be deduced from it by a limiting process. The deduction for a wire source is carried out in detail in the theoretical part below.

Next to source assumptions, we will also make a few general assumptions regarding the deposit. Since we shall be concerned with the thickness of the deposit and its optical, electrical or other consequence, we assume for the sake of simplicity that the density of the deposit is that of the bulk material. The necessary corrections can, of course, be easily made if required.

Density variations are usually due to a number of distinct phenomena associated with one or more of the following effects:

(a) A form of granulation or orientation which depends on the nature of the substrate, its temperature and the intensity or rate of deposition.

(b) Granulation or orientation of the structure of the deposit due to the angle of incidence.

(c) Chemical action in the film either at the surface or in depth e.g. oxidation during or after deposition.

Effect (b) is very important as it may invalidate some conclusions in this paper. Thus, for example, in the case of aluminium films of usual thickness (of the order of 0.1 μ) distinct granulation effects due to angle of incidence are observed at angles greater than about 60° to the normal.

A similar critical angle has been observed for platinum films and is expected to occur for most other substances. For more details in this connection see Koenig and Helwig[6] and Holland[7-8].

It should be clear that for the purposes of this paper we have assumed that the pressure of the residual gas is negligibly low and has no effect on the deposit.

THEORETICAL APPROACH

Point Source

Consider a small sphere dS_1 evaporating material at an equal rate of m g./sec. *in all directions*. Such an evaporating source will be called a point source. The amount of material passing through a solid angle $d\omega$ in any direction in unit time (see Fig. 2) is given by :

$$dM = \frac{m}{4\pi} d\omega \qquad (1)$$

Surface Source Following the Cosine Law of Knudsen

Consider a small area dS_1 from which material is evaporated at the rate of m g./sec. from one side. Then following the cosine law the amount of material passing through the solid angle $d\omega$ in a direction forming an angle φ with the normal to the surface in unit time (see Fig. 2) is given by :

$$dM = \frac{m}{\pi} \cos \varphi \, d\omega \qquad (2)$$

The results (1) and (2) show the fundamental difference between the point source and the directed source assumptions respectively. It is obvious that one really only needs the concept of a directed surface. The point source assumption then follows from it by considering a source in the form of a small sphere or part of a sphere, as discussed in the introduction.

The Mass and Thickness of a Deposit

If the material arrives at a small area dS_2 on a surface inclined at an angle θ to the direction of the vapour stream (see Fig. 3), then we can calculate the thickness and the amount of material deposited on such an area.

We have

$$d\omega = \frac{\cos \theta}{r^2} \cdot dS_2$$

so that material arrives on dS_2 at the rates

$$dM = \frac{m}{4\pi} \cdot \frac{\cos \theta}{r^2} \cdot dS_2 \qquad (3)$$

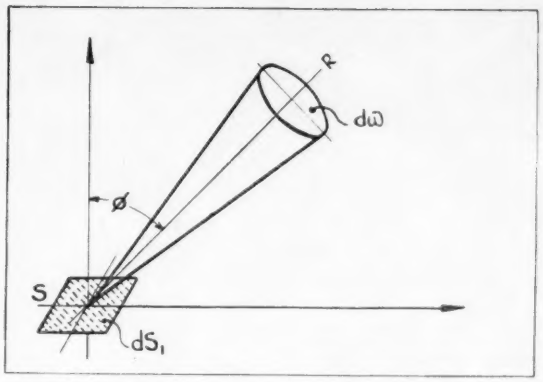


Fig. 2. Evaporation of m g./sec. from a source S through the solid angle $d\omega$ in the direction SR.

in the case of the point source, and

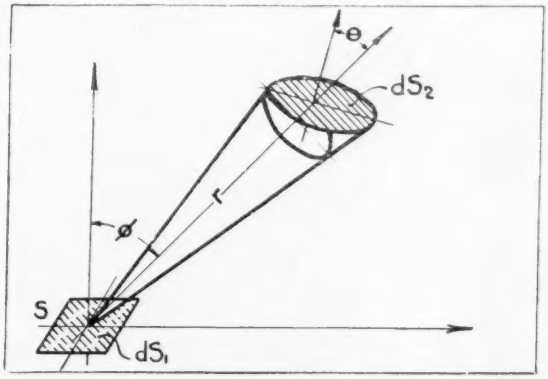
$$dM = \frac{m}{\pi} \cos \varphi \cos \theta \, dS_2 \qquad (4)$$

in the case of a directed source respectively.
Suppose the material to be evaporated has a density δ (g./cm³), and the thickness of the film condensed per unit time is t (cm./sec.), then the volume of material deposited on dS_2 is $t \cdot dS_2$, so that

$$dM = \delta \cdot t \cdot dS_2$$

The thickness of the deposit at points corresponding to the area dS_2 (see Fig. 3) is therefore given by

Fig. 3. Receiving surface dS_2 whose normal makes an angle O with the direction of the vapour stream.



the expression :

$$t = \frac{m}{4\pi\delta} \cdot \frac{\cos\theta}{r^2} \qquad (5)$$

and

$$t = \frac{m}{\pi\delta} \cdot \frac{\cos\varphi \cdot \cos\theta}{r^2} \qquad (6)$$

for the point and directed source respectively.
(5) and (6) are the basic equations for all further calculations.

The following arrangements of sources and receiving surfaces are important in practice and the distribution of the deposit has therefore been calculated. The results are given and the distribution has been plotted.

In the rest of this paper we shall eliminate the time element by taking m (g.) to be the total mass evaporated in place of the mass evaporation rate m (g./sec.) so far considered. Also t (cm.) will then be taken as the total thickness of the deposit in place of the growth-rate t (cm./sec.)

Calculation of Distribution of the Deposit

Evaporation from a Small Area onto a Parallel Plane Receiving Surface. The thickness at R follows from (6) (see Fig. 4)

$$t = \frac{m}{\pi\delta} \cdot \frac{\cos^2\theta}{r^2} = \frac{mh^2}{\pi\delta r^4} = \frac{mh^2}{\pi\delta(h^2+\rho^2)^2} \qquad (7)$$

The thickness of the deposit just below the source, at 0 is

$$t_o = \frac{m}{\pi\delta} \cdot \frac{1}{h^2} \qquad (8)$$

so that the variation in thickness is :

$$\lambda = \frac{t}{t_o} = \frac{h^4}{(h^2+\rho^2)^2} = \frac{1}{(1+(\rho/h)^2)^2} \qquad (9)$$

Evaporation from a Point Source onto a Plane Receiving Surface. In Fig. 4 we have

$$t = \frac{m}{4\pi\delta} \cdot \frac{\cos\theta}{r^2} = \frac{mh}{4\pi\delta r^3} = \frac{mh}{4\pi\delta\,(h^2+\rho^2)^{\frac{3}{2}}} \qquad (10)$$

$$t_o = \frac{m}{4\pi\delta} \cdot \frac{1}{h^2} \qquad (11)$$

and

$$\lambda = \frac{t}{t_o} = \frac{h^3}{(h^2+\rho^2)^{\frac{3}{2}}} = \frac{1}{(1+(\rho/h)^2)^{\frac{3}{2}}} \qquad (12)$$

A graph of λ against ρ/h for the two cases (9) and (12) is given in Fig. 5.

Deposits with Uniform Distribution

Arrangement of Receiving Surface to Obtain a Uniform Deposit from a Directed Surface Source of Small Area.

Consider the source as part of the surface of a sphere. Knudsen[3] has shown that the inside is uniformly coated if the cosine law is assumed (in fact he used such an arrangement to prove the cosine law experimentally by evaporating mercury on the inside of the cooled surface of a sphere where it is condensed by the first impact).

We can easily show that this type of arrangement gives a uniform deposit on the basis of this concept. Consider the plane formed by the source S a point R on the surface of the sphere and the centre O (see Fig. 6).

From Eqn. 6 the thickness at R is given by

$$t = \frac{m}{\pi\delta} \cdot \frac{\cos^2\theta}{r^2}$$

but

$$r = 2R\cos\theta,$$

so that

$$t = \frac{m}{4\pi\delta} \cdot \frac{1}{R^2} \qquad (13)$$

This is seen to be independent of the angle θ, and so, since R is fixed for any sphere, we obtain a uniform deposit with such an arrangement.

In the Case of a Point Source it is obvious that a uniform deposit is obtained on the inside of a sphere if the source is situated at the centre (see Fig. 7).

The thickness at R is again

$$t = \frac{m}{4\pi\,\delta\,R^2} \qquad (13)$$

with this arrangement.

A Thin Plane Strip Source, the long axis of which is parallel to the receiving plane and the normal of which forms an angle α with the normal to the receiving surface is shown in Fig. 8.

Here we have

$$dm = \frac{m}{l}\,d\xi$$

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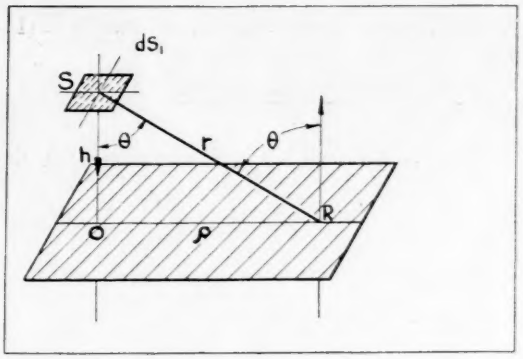

Fig. 4. Evaporation from a small area at S onto a parallel plane surface.

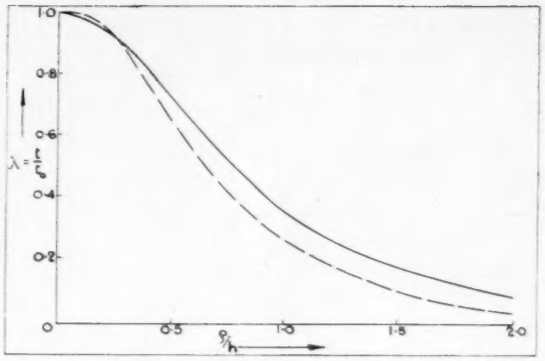

Fig. 5. Distribution of deposit on a plane surface for : (*a*) Evaporation from a small directed surface source (dotted line) (*b*) evaporation from a point source (full line).

$$dt = \frac{m}{l} \cdot \frac{d\xi}{\pi\delta} \cdot \frac{\cos\theta \cos\varphi}{r^2}$$

$$\cos\theta = \frac{h}{r}, \cos\varphi = \frac{h\cos\alpha + y\sin\alpha}{r},$$

$$r^2 = h^2 + \rho^2 = h^2 + \xi^2 + y^2$$

so that

$$t = \frac{m}{l} \cdot \frac{h(h\cos\alpha + y\sin\alpha)}{\pi\delta} \int_0^l \frac{d\xi}{(h^2 + y^2 + \xi^2)^2}$$

which with $a^2 = h^2 + y^2$ gives:

$$t = \frac{m}{l} \cdot \frac{h(h\cos\alpha + y\sin\alpha)}{2a^2\ \pi\delta} \left(\frac{l}{a^2 + l^2} + \frac{\tan^{-1}\left(\frac{l}{a}\right)}{a} \right) \quad (14)$$

In the more general case, (shown in Fig. 9) of a strip lying symmetrically to the z-axis, and a general point $R \equiv (x, y)$ on the receiving plane, we have the thickness at R given by:

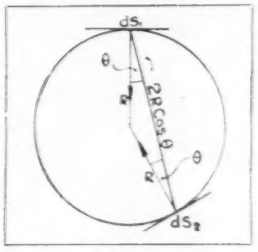

Fig. 6. Evaporation onto the inside of a sphere from a surface source dS_1 situated at the circumference.

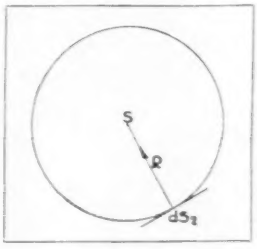

Fig. 7. As Fig. 6 but a point source at the centre.

$$t = \frac{m}{l} \cdot \frac{h(h\cos\alpha + y\sin\alpha)}{2a^2\pi\delta} \left(\frac{\frac{l}{2} + x}{a^2 + \left(\frac{l}{2} + x\right)^2} + \frac{\frac{l}{2} - x}{a^2 + \left(\frac{l}{2} - x\right)^2} + \frac{1}{a}\tan^{-1}\left(\frac{\frac{l}{2} + x}{a}\right) + \frac{1}{a}\tan^{-1}\left(\frac{\frac{l}{2} - x}{a}\right) \right) \quad (15)$$

or

$$t = \frac{m}{l} \cdot \frac{h(h\cos\alpha + y\sin\alpha)}{2a^2\pi\delta} \left(\frac{l\left(a^2 - x^2 + \frac{l^2}{4}\right)}{(a^2 + x^2)^2 + \frac{l^2}{2}(a^2 - x^2) + \frac{l^4}{16}} + \frac{1}{a}\tan^{-1}\left(\frac{la}{a^2 + x^2 - \frac{l^2}{4}}\right) \right) \quad (16)$$

and clearly to O we have :

$$t_0 = \frac{m}{l} \cdot \frac{\cos\alpha}{2\pi\delta} \left\{ \frac{l}{h^2 + \frac{l^2}{4}} + \frac{1}{h} \tan^{-1} \frac{lh}{h^2 - \frac{l^2}{4}} \right\} \quad (17)$$

The bracket expressions in (14), (15) or (16), and (17) will be denoted by A, B and C respectively for future reference.

The result in this form is too general to be of direct use, but we can deduce other important practical results from it. For instance consider the *Source Strip to Lie Parallel to the Receiving Plane.* We then have $\alpha = o$, so that

$$t = \frac{m}{l} \cdot \frac{h^2}{2a^2\delta\pi} \cdot \left\{ B \right\} \quad (18)$$

where B is the bracket expression in (15) or (16) and

$$t_0 = \frac{m}{l} \cdot \frac{1}{2\pi\delta} \cdot \left\{ C \right\}$$

where C is the bracket expression in (17).
$\lambda = t/t_0$ *along the x axis* is plotted in the graph shown in Fig. 10.

A Cylindrical Evaporation Source

Eqn. 17 for the inclined plane source can also be used to deduce the distribution of the deposit coming from a *cylindrical source.* The axis of the cylinder is again assumed to lie parallel to the receiving surface as shown in Fig. 11.

We have $dm = (m/2\pi)\, d\alpha$ so that for the plane strip forming part of the cylinder, we have as in (14) above:

$$dt = \frac{m}{4\pi^2} \cdot \frac{d\alpha}{\delta} \cdot \frac{h'(h'\cos\alpha + y'\sin\alpha)}{l(h'^2 + y'^2)} \cdot \left\{ A' \right\}$$

where A is the bracket expression in (14), the dash indicating that

$$h' = h - \rho\cos\alpha,\ y' = y - \rho\sin\alpha$$

has been taken. Also

$$\beta = \cos^{-1}\frac{\rho}{a},\ \Psi = \tan^{-1}\frac{y}{h} \text{ where } a^2 = y^2 + h^2$$

The effect of the whole cylinder would then be obtained from

$$t = \frac{m}{4\pi^2\delta l} \int_{\alpha = \Psi - \beta}^{\alpha = \Psi + \beta} \frac{h'(h'\cos\alpha + y'\sin\alpha)}{h'^2 + y'^2} \cdot \left\{ A' \right\} d\alpha \quad (19)$$

It is beyond the scope of this paper to calculate this and we turn now to the more important practical case of a thin wire.

Evaporation from a Thin Straight Wire on to a Parallel Plane Receiving Surface. The wire being thin, we can take $\rho \to 0$, so that $h' = h$, $y' = y$. Therefore $A' = A$ and then

$$t = \frac{m}{4\pi^2\delta l} \cdot \frac{h}{a^2} \cdot \left\{ A \right\} \cdot \int_{\Psi - \beta}^{\Psi + \beta} (h\cos\alpha + y\sin\alpha) d\alpha$$

$$= \frac{m}{4\pi^2\delta l} \cdot \frac{h}{a^2} \cdot \left\{ A \right\} \cdot 2\sin\beta\,(h\cos\psi + y\sin\psi)$$

$$t = \frac{m}{2\pi^2\delta l} \cdot \frac{h}{a} \cdot \left\{ A \right\} \cdot \sin\beta$$

Also clearly for a *thin* wire $\sin\beta \to 1$, so that finally we have

$$t = \frac{m}{2\pi^2\delta l} \cdot \frac{h}{a} \cdot \left\{ A \right\} \quad (20)$$

with A given by the bracket in (14) above.

Clearly one can again consider the more general case given in

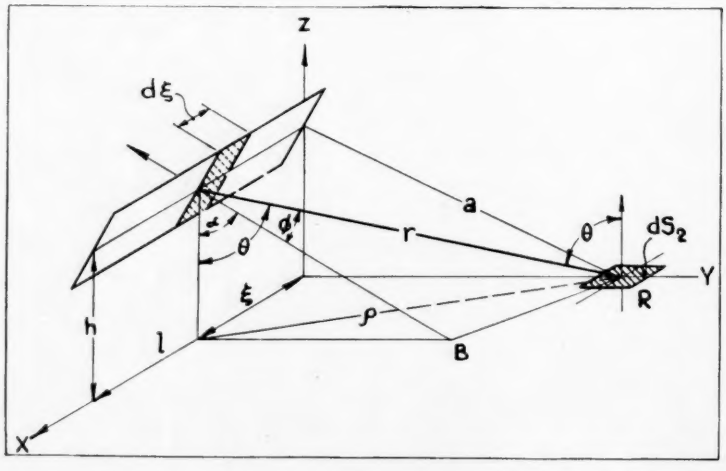

Fig. 8. Plane strip source inclined to the receiving plane (x,y) by an angle α. dS_1 is a typical elementary surface source.

Fig. 9 and then

$$t = \frac{m}{2\pi^2\delta l} \cdot \frac{h}{a} \cdot \left\{B\right\} \qquad (21)$$

where B is the bracket expression in (15) or (16) above and

$$t_o = \frac{m}{2\pi^2\delta l} \cdot \left\{C\right\}$$

The distribution $\lambda = t/t_0$ along the x - axis is the same as for the case of a strip parallel to the receiving surface and is again given by the Graph (see Fig. 10). The Eqn. 21 for a thin wire closely resembles that of a directed strip as in (15) or (16) and can be obtained from this except for the factor $1/\pi$ by taking $\alpha = \psi$ in (15) or (16), *i.e.* an equivalent strip the normal of which is directed at the point R which lies on the receiving surface. Then we get

$$\cos\alpha = \cos\psi = \frac{h}{a}$$

$$\sin\alpha = \sin\psi = \frac{y}{a}$$

and $$h\cos\alpha + y\sin\alpha = a.$$

A Short Wire or an Element of a Wire Source. This can now be deduced from the result for a wire by taking $l \to O$ in Eqn. 21 with B given by (16). It is seen that

$$\lim_{l\to o} B = \frac{l(a^2-x^2)}{(a^2+x^2)^2} + \frac{l}{a^2+x^2} = \frac{2\,la^2}{(a^2+x^2)^2} \qquad (22)$$

so that the thickness of the deposit on a plane parallel to the axis of such a source element is

$$t = \frac{m}{\pi^2\delta} \cdot \frac{ha}{(a^2+x^2)^2} = \frac{m}{\pi^2\delta} \cdot \frac{ha}{r^4} \qquad (23)$$

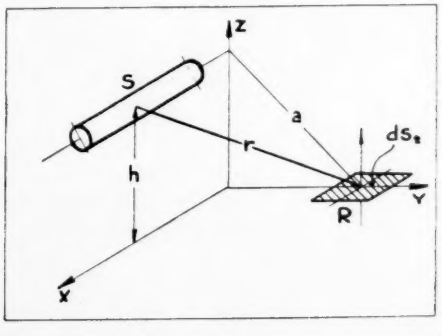

Fig. 12. Wire element (S) evaporation source with its axis parallel to the x-axis.

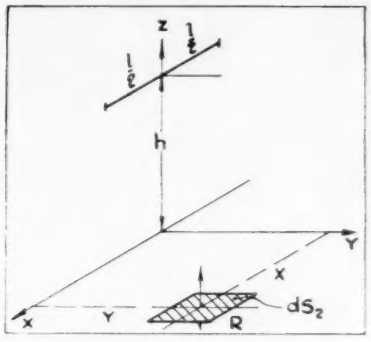

Fig. 9. Arrangement of the receiving point R relative to the strip source. R is taken to be a general point in the x-y plane with the strip source lying centrally and symmetrically to the z-axis.

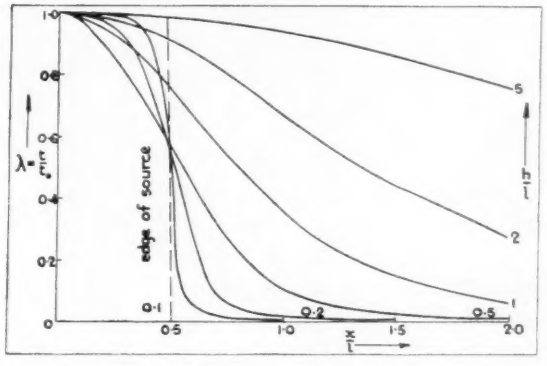

Fig. 10. Distribution on a plane surface when evaporating from a strip or wire source arranged as in Fig. 9. The decrease in thickness of deposit for points on the x-axis is shown for various distances from the source.

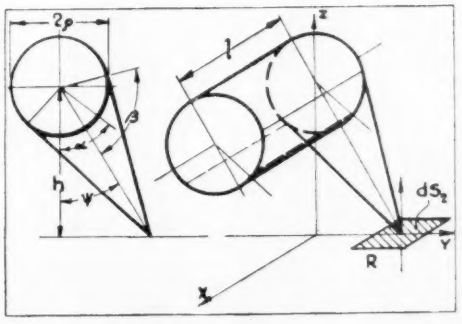

Fig. 11. Cylindrical evaporation source length l, radius ρ with axis parallel to the receiving plane.

The respective symbols are explained in the diagram in Fig. 12. The result given in Eqn. 23 can be added to the fundamental equations to form a starting point for the consideration of more complicated source constructions in wire form.

Ring Sources

A simple arrangement which is of very great practical importance for obtaining nearly uniform deposits on relatively large areas consists in either a number of small sources of the surface- or point-type arranged in a circle parallel to the receiving surface, or in a single small source evaporating onto a rotating plane receiving surface so that in effect with respect to that surface one has a continuous ring of surface- or point-sources. Taking the latter case the distribution of the deposit has been worked out and it has been found that under certain conditions an almost uniform deposit is obtained over an appreciable area.

Consider then an arrangement as shown in Fig. 13. We have $dm = (m/2\pi)\, d\varphi$ in the case of continuous ring sources. Also from the triangle in Fig. 13(b):

$$r^2 - h^2 = R^2 + A^2 - 2AR\cos(\pi-\varphi)$$

so that we get for r the result :

$$r^2 = R^2 + A^2 + h^2 + 2AR\cos\varphi$$
$$= h^2 + (R+A)^2 - 4AR\sin^2\frac{\varphi}{2}$$

In the Case of a Continuous Ring of Small Surface Sources (parallel to receiving plane) we have from Eqn. 7

$$dt = \frac{h^2 dm}{\delta\pi r^4} = \frac{m}{2\delta\pi^2}\cdot\frac{h^2}{r^4}\cdot d\varphi$$

and hence,

$$t = \frac{mh^2}{2\delta\pi^2}\int_0^{\pi}\frac{2\,d\,\varphi}{\left(h^2+(R+A)^2-4AR\sin^2\frac{\varphi}{2}\right)^2}$$

Which can be shown to give

$$t = \frac{mh^2}{2\delta\pi}\cdot\frac{2h^2+(A+R)^2+(A-R)^2}{(h^2+(A+R)^2)^{\frac{3}{2}}(h^2+(A-R)^2)^{\frac{3}{2}}}$$

or

$$t = \frac{mh^2}{\delta\pi}\cdot\frac{(h^2+A^2+R^2)}{(h^2+A^2+R^2+2AR)^{\frac{3}{2}}(h^2+A^2+R^2-2AR)^{\frac{3}{2}}} \quad (24)$$

The thickness at the origin is obviously then:

$$t_o = \frac{mh^2}{\delta\pi}\cdot\frac{1}{(h^2+R^2)^2} \quad (25)$$

From these results, t has been plotted against A for various values of h with R = 1 as shown in Fig. 14(a). The region of interest for obtaining very uniform deposits for accurate work is shown plotted on a larger scale in Fig. 14(b).

We now consider the case of a *Continuous Ring of Point Sources*. In this case we have from Eqn. 10

$$dt = \frac{hdm}{4\pi\delta r^3} = \frac{m}{8\pi^2\delta}\cdot\frac{h}{r^3}\,d\varphi$$

and, hence,

$$t = \frac{mh}{8\pi^2\delta}\int_0^{\pi}\frac{2d\varphi}{\left(h^2+(R+A)^2-4AR\sin^2\frac{\varphi}{2}\right)^{\frac{3}{2}}}$$

with $\varphi/2 = \theta$ we get,

$$t = \frac{mh}{8\pi^2\delta}\cdot\frac{4}{(h^2+(R+A)^2)^{\frac{3}{2}}}\int_0^{\pi/2}\frac{d\theta}{(1-k^2\sin^2\theta)^{\frac{3}{2}}}$$

where $k^2 = 4AR/(h^2+(R+A)^2)$ so that we get an elliptic integral in one of the standard forms as given in ' Tables of Functions with Formulae and Curves '[15]. The integral is then $E(k, \pi/2)\cdot 1/(1-k^2)$, so that we get finally writing $E(k, \pi/2) \equiv E(k)$, for the thickness

$$t = \frac{hm}{2\pi^2\delta}\cdot\frac{E(k)}{(h^2+(A+R)^2)^{\frac{1}{2}}(h^2+(A-R)^2)} \quad (26)$$

which is in agreement with the result described in ' Modern Physical Laboratory Practice ' [13], of Fisher and Platt[26] and that discussed recently by Prugne and Leger[14]. Values of this have been calculated and are plotted in the graph shown in Fig. 15 with R = 1.

Comparison of the results plotted in Figs. 14 and 15 immediately shows a most remarkable similarity in the form of the distribution of the deposit. In both cases a fairly uniform film is obtained when h is approximately equal to the radius of the ring, the uniformity extending almost over the complete diameter. Strong has shown (*loc.cit.*) how this principle can be extended to larger surfaces by using sources

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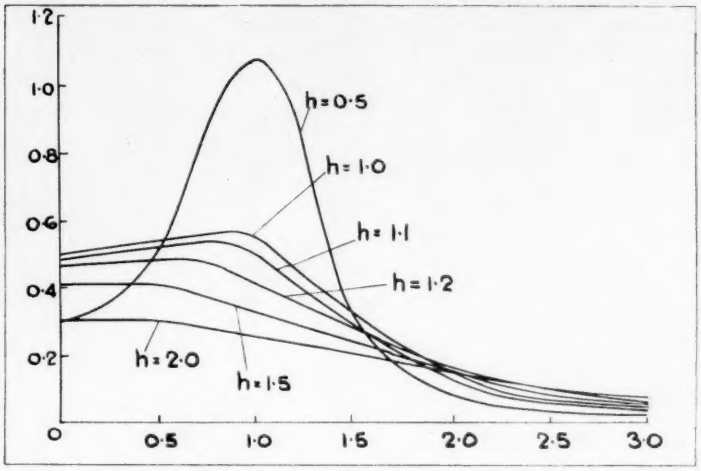

Fig. 14(*a*). Distribution of deposit on a plane surface when evaporating from a ring of surface sources. A is plotted against $t \cdot \frac{2\delta\pi}{m}$ with R = 1.

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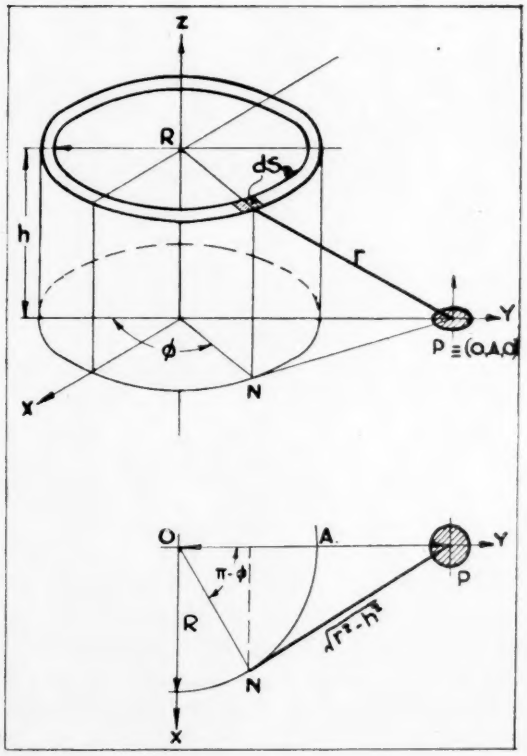

Fig. 13. Ring source radius R parallel to the receiving plane. (*a*) General arrangement, (*b*) detail on receiving plane (x,y).

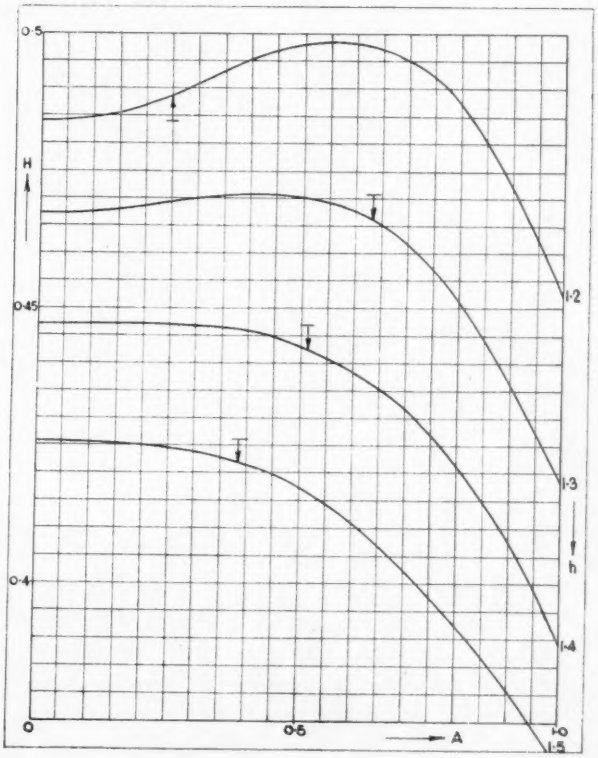

Fig. 14(*b*). Detail of graph above showing uniform part enlarged.

arranged in concentric rings. It is obvious that a similar extension is possible for the surface-type source. Since the two extreme types of sources, namely the directed-surface source and the point source, give nearly identical results when arranged as continuous rings, it is to be expected that sources intermediate between these two, such as short wires, spirals, etc. will also give similar results. This has actually been borne out in practise and has often been wrongly regarded as a practical proof that such sources behave like point sources.

EXPERIMENTAL VERIFICATION

Introduction

There are two ways of verifying the above assumptions and results:

(1) the amount of material deposited at a certain point can be checked to see whether it is of the order expected theoretically and

(2) one can measure the falling-off in thickness and density of the deposit over a certain area to see that it agrees with that indicated by the formulae and curves.

Both methods have been used extensively with great success in a number of decisive experiments, some of which will be described. Generally speaking, method (1) is the more definite as shown, for instance, by comparison of Eqns. 8 and 11. Thus, if allowance is made in Eqn. 11 for evaporation from the point source over only half a sphere, these equations show that the ratio of thickness of deposit for the two types of sources under identical geometrical conditions is 2. This factor should be easily detected, but on the other hand, the absolute value of density or thickness is much more difficult to obtain with certainty in practice. This difficulty will not arise to the same extent in method (2), since the density or thickness uncertainty only introduces a constant though possibly unknown factor for each material. In method (2) the difference in density or thickness variation for the two assumptions is not so marked but it can nevertheless be demonstrated. Also there is the difficulty that the density or thickness variations are not easily measured to a high degree of accuracy. These points will be discussed in greater detail in the particular experiments to be described.

Magnesium Fluoride Experiment

Dielectric films from materials such as magnesium fluoride or zinc sulphide for optical interferometry are often evaporated from refractory metal strips bent into the form shown in Fig. 1(e) with a small circular dimple at the centre for holding the evaporant. Vapour source heaters of this kind have been in use for some years, particularly for anti-reflection coating. They are easily classified as being of the surface type, yet for some reason their vapour emission has been commonly and erroneously described as that of a point source.*

An experiment was carried out with magnesium fluoride to check the surface-source concept by method (1) and is described in the following paragraphs.

Several small glass plates were thoroughly cleaned and arranged normally at various distances above a plane source of the type shown in Fig. 1(e). A highly pure MgF_2 specially prepared by British Drug Houses Ltd. for lens coating was placed in the source and initially degassed and fused at a low pressure. The heater strip with the evaporant fused to its surface was then weighed. The glasses were further cleaned by ionic bombardment and films of suitable thickness were then deposited. After the evaporation the heater strip and remaining evaporant were again weighed to determine the total mass evaporated. The film thickness was measured by the multiple-beam interferometry technique of Tolansky.[17]

In Table I the observed thickness values are shown together with the thickness values calculated from the Knudsen surface emission formula (Eqn. 8) for evaporant charges of different masses and varying source distances. To compare these measurements with the calculated values one has to assume a density value for the deposited film. The thickness calculations are given for values of density based on the bulk density of MgF_2 $\delta = 3$ g./cm.^{3} and also that given by Rood [18], for unbaked MgF_2 films, $\delta_f = 2.56$ g./cm.^{3}.

e.g. Davey[16], in a recent work on deposition techniques, states that this type of source has point source distribution.

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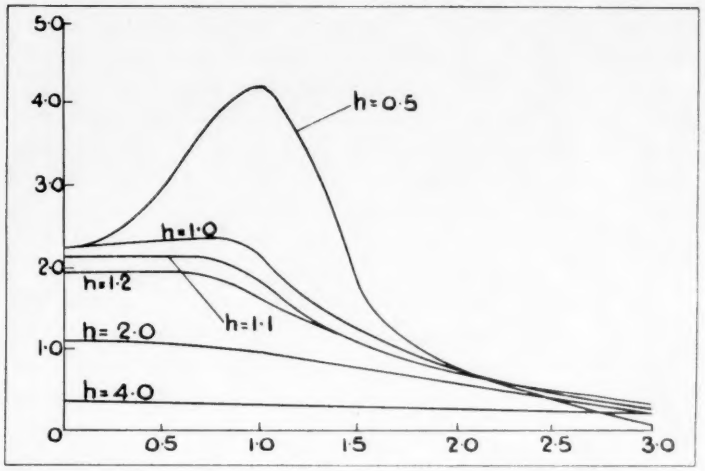

Fig. 15. Distribution of deposit on a plane surface when evaporating from a ring of point sources. A is plotted against $t \cdot \frac{8\pi^2\delta}{m}$ with R = 1.

Rood arrived at this density value by directly weighing the evaporated films and calculating the true thickness from the optical path length.

Comparison of the results in Table I shows that, to a first approximation, the assumption of source emission according to the Knudsen law is in good agreement with the observed results, even if there is some uncertainty with regard to the density values.

It is, in fact, of interest to note that the calculated thickness values based on bulk density are closer to the observed values than are those based on the density given by Rood. Whilst it is true that the film structure of evaporated deposits may vary considerably, being dependent on complex formation conditions, there can be no doubt that films of MgF_2 have a lower density than the bulk material. The slight discrepancy in the results could be explained by a certain degree of source collimation but this is uncertain because there is some doubt as to what is in fact measured by multiple-beam interferometry. Thus Avery[19] found that the measured thickness values may vary considerably with the nature of the metal used for the reflecting layer on top of the film under study. He contended that this was due to differences in the molecular packing of the layers, but his results were not confirmed by Heavens[20]. Undoubtedly there must be some tendency for vapour atoms to penetrate the grain interstices during the deposition of the reflecting layers (silver was used in our experiments) so that the thickness measured should be between the average and the total value as given in Table I.

Table I. **Strip Source Experiment with MgF_2**

Source to Specimen Distance (cm.)	*Calculated Thickness (Å)*		*Observed Thickness (Å)*
	δ=2.56 *g./cm.*^{3}	δ=3.0 *g./cm.*^{3}	
20	1950	1660	1680
20	1790	1525	1430
15	4590	3915	4050
20	2580	2200	2310
25	1650	1410	1470

Zinc Sulphide Experiment

The use of a surface emitting source for ZnS is of considerable practical interest because it is widely used in the deposition of optical interference films.

Zinc sulphide powder was compressed into the form of a pellet and then evaporated onto glass using a similar evaporation technique to that described for magnesium fluoride.

The reflectivity of the ZnS films as a function of wavelength was measured and from this the optical thickness of the film could be found.

An approximate value of film thickness was then obtained by dividing the optical thickness n t by the refractive index n of bulk ZnS.

Comparing the mass required to form a film of a certain specified thickness with that calculated in accordance with the surface emission concept, it was discovered that the calculated mass differed by a factor of 0.3 - 0.5. This was confusing, especially as a previous investigation by Holland and Newman[21] of the general film distribution of evaporated ZnS on a spherical holder (*i.e.* an investigation of type (2) as discussed above) had shown that the deposit was consistent with the surface emission assumption. Further experiments described below in another connection confirmed these results.

Let us therefore briefly consider the possible reasons for the increased mass of ZnS required to form a film of a certain thickness :

(1) The evaporant partially decomposes during the evaporation.
(2) The adsorbed water is not removed during the preliminary degassing bake.
(3) A percentage of the vapour molecules arriving on the surface does not condense.
(4) Some vapour molecules are scattered in transit and prevented from arriving at the condensing surface.

Critical Discussion and Subsidiary Experiments on ZnS Films

Rood[25] has reported that rapidly evaporated ZnS films have a high light absorption and claims that the deposition rate for producing good quality films should be about 100 Å/min. Slowly evaporated ZnS films are very soft and thus generally a compromise is made to obtain both durable and non-absorbing layers. Polster[22] confirming Rood's results found that rapidly deposited ZnS films (1,000Å/min.) had considerable light absorption. He claims that large ZnS crystals are formed at high deposition rates which increases the amount of light lost by scattering within the film. This would be the reverse to general experience where it is found that the grain size of most substances falls as the rate of deposition increases.

The greater light absorption of rapidly deposited ZnS films could be explained by decomposition of ZnS during evaporation.

It is known that zinc sulphide is partially decomposed into hydrogen sulphide and zinc oxide when raised to a high temperature in the presence of steam[24]. There is an ample supply of water vapour to produce such a reaction during evaporation because samples of ZnS powder used in our tests showed a weight reduction of some 30% after baking at high temperature to remove water vapour. Further, the residual atmosphere in a kinetically pumped vacuum system is mainly water vapour liberated from the walls of the vacuum chamber. There was in fact some evidence for the appearance of sulphur compounds, both in the rotary pump and the vacuum chamber.

The decomposition of zinc sulphide would have different effects depending on the rate of evaporation. At high rates of evaporation free zinc atoms may be trapped in the condensed film, whereas at low rates there is ample opportunity for the zinc to oxidise either at the condensing surface or in the region of the source. Zinc oxide formed in the source would not evaporate because of its low volatility. Zinc oxide has a refractive index comparable to that of ZnS and would not be easily detected optically if present in the deposit.

Although decomposition is a very likely explanation of these light absorbing effects—a subject which calls for further investigation—it was concluded that this was not the cause in our case, as such great evaporant losses would have required either the formation of highly absorbing layers or the formation of almost pure ZnO films.

Similarly the possibility of water vapour loss must also be ruled out as an explanation of the observed effect, although it undoubtedly takes place to a small extent. This was shown conclusively from the results of the following experiments.

It was argued that, if there was decomposition or loss of weight due to evaporation of adsorbed water, then this would not be a specific property of any particular source geometry or design. Experiments were therefore carried out using a basket source of the type shown in Fig. 1(b). A weighed quantity of carefully degassed and sintered ZnS powder sufficient to form a quarter wavelength film for green light (assuming point source emission, *i.e.* $t = M/4\pi r^2\delta$) was

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evaporated. The glass slide specimens were arranged to receive vapour molecules at normal incidence when lying on the circumference of a circle with the source at the centre. Slide S_1 and S_2 were placed vertically one above and one below and S_3 horizontally. In this way the degree to which vapour emission approached the pattern of a point source could be studied. From the reflectivity measurements for the ZnS film deposit on each slide the optical thickness and hence the film thickness was determined as usual assuming a refractive index $n = 2.3$.

The results in Table II show that no losses occurred in this case and in fact a quarter-wavelength film was deposited on each slide. Hence, for a tungsten basket heater both the intensity distribution and the film thickness deposited is fairly accurately given by calculations based on point source emission. This proved that no appreciable losses either by decomposition or evaporation of water vapour could have taken place in the source itself. The other remaining possible explanations, namely scattering or non-condensation of the vapour could have been easily masked in this experiment due to the uniformity and symmetry of the arrangement.

Table II. **Point Source Experiment With ZnS** (Basket Type Heater) Refractive Index Taken as $n = 2.3$

Slide Position	*Measured Film Thickness* (Å)	*Calculated Film Thickness* (Å)
S_1	710	580
S_2	670	
S_3	460	

Further tests showed that the increased evaporant mass required for a given thickness film when evaporated from a Knudsen surface source could not be explained by the phenomen mentioned under (3) on page 358. It was shown that the losses persisted when the receiving surface was coated with a very thin layer of silver in order to alter the surface forces and aid condensation.

We still have to discuss the effect of scattering of vapour molecules by collisions. We will distinguish between two ways in which scattering can occur. First there is the possibility of scattering due to collisions between the vapour molecules themselves and secondly scattering due to collisions between vapour molecules and gas molecules. The former phenomenon is associated with high evaporation rates and would be most noticeable in the vicinity of the source. There the vapour density is greatest so that the source is surrounded by a ' cloud ' of vapour, the boundary of which is defined by the region in which the fall-off in vapour density (or the increase in mean free path) is sufficient to allow for neglecting any further vapour-vapour scattering.

In the region beyond the cloud the vapour can then be considered as issuing from the cloud rather than from the source and this would obviously affect the calculations. Such scattering can in fact occur and is easily demonstrated by placing a small slide a few centimetres from the source. If the vapour intensity is increased or the transit distance is small, the receiving surface is engulfed in the cloud and a thick condensed deposit is able to form on the surface not facing the source. At the specimen distances and evaporation rates actually used in our tests only a negligible film of zinc sulphide was condensed on remote surfaces. Thus it can be presumed that, if vapour-vapour molecular scattering took place, it would be confined to a small part of the transit path.

On the other hand scattering due to residual gas molecules would be fairly constant throughout the vapour path, depending of course on the pressure in the evaporation plant.

Experiments were therefore made to investigate these possibilities and their effect on the observed evaporation losses from the Knudsen surface source.

The evaporation rate for a strip heater was first decreased to a very low order so that the ZnS film formation rate was only 15 Å/min. This should greatly decrease the vapour - vapour molecular collisions in transit. It was found that the evaporant losses remained constant. This points to the fact that the losses cannot be explained by scattering in this way.

If any appreciable cloud formation existed in the vicinity of the source, then this should affect the distribution of the deposit; the presence of a cloud would tend to nullify the directional properties of the source and make it appear more like a point source.

This was tested by mounting several glass slides in a ring holder of 22.8 cm. diameter, evaporations being made with a strip source mounted first at the centre of the radius of curvature and secondly at the circumference of the holder. The thickness of the deposit on each slide was determined as before by measuring reflectivities and the results are given in Table III. The position of each slide is given in terms of its angle measured from the centre and against the vertical. In each evaporation the mass of ZnS evaporated was sufficient to deposit an approximately half-wavelength film on the slide directly above the source. Examination of the results shows that the more uniform deposit is produced with the vapour source on the holder circumference.

Table III. **Film Thickness Distribution For ZnS**

	Film Thickness in Å		
Angular Position Of Slide. Measured From Vertical	*Boat At Centre Of Circle. Evaporation Time:* 15 *mins.*	*Boat At Circumference Of Circle. Evaporation Time:* 20 *mins.*	*'Pepperpot' Boat On Circumference Of Circle. Time:* 10 *mins.*
−157	55	350	–
−135	95	400	770
−112	0	380	710
−90	0	400	690
−67	150	400	680
−45	310	420	680
−22	–	420	730
0	400	420	710
45	460	420	710
90	175	420	710
135	0	–	710
157	0	350	–
180	55	–	–

The quantity evaporated was sufficient in each case to deposit a calculated thickness of 1,100 A for the angular position O *i.e.* with the slide normal to the source) on the basis of a surface emitter.

Thus, in spite of the considerable losses of evaporant the intensity distribution is that of a strip source. An interesting feature of the slides coated with the source at the centre of the holder is that a slightly scattered deposit had formed on the glass slides which were not directly facing the vapour source, *i.e.* those glasses that were mounted on the lower half of the holder.

These experiments were than repeated by using a surface source constructed on the 'pepperpot' principle by completely covering the ZnS material on the strip heater with a shield containing 5 × 5 fine holes, 0.2 mm. diameter in a regular array 1 mm. apart. The results with this source are listed in the last column of Table III. It can be seen that the distribution is still uniform and the losses have been very substantially reduced.

Our final conclusions from these experiments are that we are here dealing with a case of gas scattering due to the usual residual gas pressure (10^{-5} to 10^{-4}mm. Hg) and accentuated by the low velocity of the vapour stream. The question arises, why this effect is not normally noticed with other substances evaporated at this pressure level. It is in fact not usually found and the reason for finding it with ZnS is :

(a) Due to the strong inclination for decrepitation the evaporation rate is normally kept very low when using open crucibles.

(b) The vapour pressure is rather high and this means an unusually low evaporation temperature.

In addition, it should be noted that ZnS is in any case difficult to heat because of its low heat conductivity and poor thermal contact with the heater. The 'pepperpot' construction facilitated the achievement of a higher ZnS temperature and therefore a higher molecular velocity and this explains, why this construction gave a considerable increase in the total amount deposited.

Further experiments will of course be required to substantiate this claim. Providing this can be proved these preliminary results would imply that the effect of gas scattering at these low pressures is :

(a) To deflect a proportion of vapour molecules away from the receiving surfaces.

(b) The proportion of vapour so deflected depends on the velocity of the vapour stream emitted from the source (being highest at low source temperatures).

(c) The vapour atoms arriving on the receiving surface have on the whole not been scattered because a high proportion of these have not suffered collisions with the gas.

The last mentioned effect would explain why the angular distribution of the deposit had not been

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appreciably disturbed and yet the total amount collected at each point was reduced.

It is clear from this discussion that a more thorough investigation of the evaporation characteristics of zinc sulphide is required before the mechanism can be fully explained.

Uniform Deposits from Directed Surface Sources

A small source emitting from its surface according to the cosine law will give a uniform deposit inside a sphere if the source is placed on the circumference (see discussion above in connection with Eqn. 13). This can be checked over a substantial part of the sphere – namely up to angles of incidence and film thicknesses for which the deposit does not become too granular. Holland and Newman[21] have recently investigated deposits inside such a spherical holder. They have also shown, by moving the source along the diameter of the sphere, that the position on the circumference did, in fact, give maximum uniformity. The substances investigated by them included zinc sulphide layers and silver. In the latter case one has an example of a substance which does not wet the source and so does not flow over the heater surface, the molten metal forming a spherical globule instead. It was surprising to find that evaporation of a spherical globule on a flat heater strip also gave a uniform deposit over a substantial part of the sphere, thus exhibiting emission properties in accordance with a Knudsen surface. One would have expected such a globule to behave like a typical point source. This phenomenon is explained by the fact that the heater surface which is at least as hot as the globule itself acts as a reflector for the emitted vapour so that a high percentage of the silver atoms emitted from the lower edges of the globule collided with the surface of the heater where they re-evaporated as from a surface.

In the paper by Ranc[12] the cosine law is also recognised and experiments are described in which this assumption is tested with gold as the material by method (1), *i.e.* giving the correct strength of deposit at different distances. He states that the source would have to be wetted to get agreement with the surface emission formula. This did not seem a necessary condition if the heater surface is also a vapour reflector as in the case of the silver investigation of Holland and Newman just discussed.

Aluminium Experiments with a Cylindrical Source

The source was of the type shown in Fig. 1(c) and consisted of three 0.5 mm. diameter tungsten wires twisted together and wound in the form of a close spiral (approx. 5 turns per cm. length and 0.4 cm. diameter). A length of 22 S. W. G. ' superpurity ' aluminium wire, obtained from Johnson Matthey & Co. Ltd., was placed axially into the spiral.

When the aluminium was pre-fused to the filament its excellent wetting properties allowed it to flow around the twisted tungsten wires filling in the interstices all along the spiral in a uniform manner. Clean glass slides were placed along a line parallel to the axis of the spiral and perpendicular to this plane. After a period of discharge cleaning some of the aluminium was evaporated but leaving a quantity on the source. The investigation was of the type designated above as method (2), *i.e.* it was concerned with the distribution of the deposit, thus no absolute measurement of thickness or density was required.

To measure the density of various points on the deposit relative to the maximum at the centre the following procedure was adopted:

The glass slides were examined optically and the reflectivity R and transmission T of the aluminium films were determined.

The absorption A (in %) is then given by the relation $A = 100 - R - T$. It was independently established that the absorption coefficient of aluminium films was reasonably constant with thickness for the range of thickness values considered in the investigation. This was done by plotting values of

$$D = \log\left(\frac{100 - R}{T}\right)$$

where D is the optical density of the film against thickness t. The transmission and reflection coefficients as a function of thickness were those given

by Walkenhorst[23]. The graph showed that over the range, we are interested in, thickness and optical density were proportional, so that we could take:

$$\lambda = \frac{t}{t_o} = \frac{\log\left(\frac{100-R}{T}\right)}{\log\left(\frac{100-R_o}{T_o}\right)} = \frac{D}{D_o}$$

Where the suffix indicates the values of the maximal position immediately below the centre of the source.

The values of D/D_o found in this way are plotted against the distance to filament-length ratio x/l. The experiment was performed with several filament lengths and source distances and the curves shown in Fig. 16 are plotted for the h/l values of 5, 2 and 1, (for the nomenclature see Fig. 9), *i.e.* source distances 25 cm., 20 cm. and 5 cm. with filament lengths 5 cm., 10 cm., and 5 cm. respectively. In the same diagram the theoretical value is plotted for a truly cylindrical wire (which is the same as in the corresponding curves of Fig. 10). Considering the measurement difficulties and approximations, the agreement between the theoretical and observed ratios is very good.

The sharp falling-off in the case of h/l values smaller than 1 was also observed but there the range of density values was too large to measure satisfactorily by this method.

The approximation of a closely wound spiral to the emission characteristics of a cylindrical surface source has therefore been demonstrated for aluminium. The same approximation should hold equally well for a great number of other metals for which the spiral wire source construction would be an advantage as for example in the case of metals evaporated from electroplated filaments (such as rhodium).

An application of these formulae and curves (Fig. 10) which comes to mind immediately lies in the construction of variable density filters which are in any case often carried out with electroplated rhodium on tungsten or nickel-chrome alloy in spiral filaments. Thus by choosing one of the curves in Fig. 10 or others obtained from the formulae, a fairly linear density fall-off can be picked out over quite a large range.

Another source design which can be expected to follow this type of distribution is the type indicated in Fig. 1(f) made from a folded sheet of refractory material. This should be suitable for powder material, but has not yet been checked for distribution.

CONCLUSIONS

The results of the experiments described in this article establish beyond doubt that the distribution of the material deposited by evaporation from strip-type heater sources is in perfect keeping with the concept of surface emission and suggests a number of possible source arrangements to obtain uniform deposits. The simplest arrangement would provide

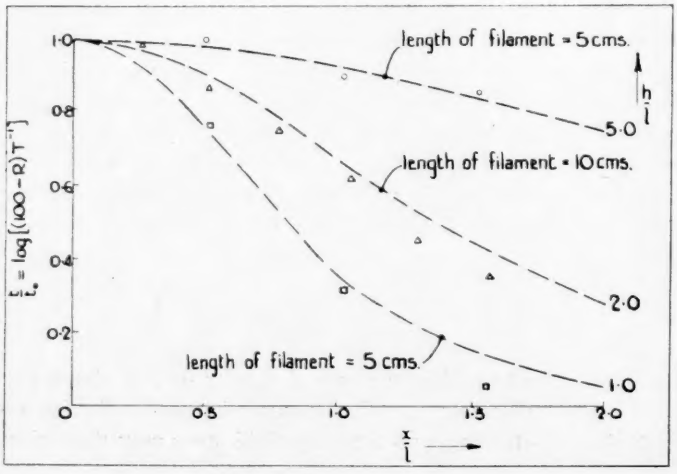

Fig. 16. Distribution of deposit on a plane surface, parallel and in the same plane as the source. The source was a helix with a diameter of 0.4 cm. made of 3 strands of 0.5 mm. diameter tungsten wire. The pitch of the helix was approximately 5 turns per cm. length of filament. Dotted lines show the theoretical distribution assuming a thin wire source. Points indicate the values obtained experimentally.

for the articles to be placed on part of a sphere with the source on its circumference. Also this concept facilitates fairly exact calculations of film thickness for some materials. Thus it was shown that MgF_2 could be deposited to a reasonably accurate thickness by weighing the amount of material in the source and this procedure may be of value in making anti-reflection films where conditions render it difficult to observe the coated surface.

In applying the results of this investigation, however, one should carefully examine conditions in each case. There are a number of factors which have to be taken into account. For instance, weight determinations are not always reliable. A typical example of this was given in the discussion on the experiments with zinc sulphide. As stated before the case of zinc sulphide requires additional research work.

It is shown that tungsten spirals have emission properties approximately the same as cylindrical wire sources.

In the experiments described we have restricted ourselves to the use of optical methods for the determination of thickness and density. It may be argued that there are more accurate methods, such as weighing the mass of the deposit on a given area by means of a microbalance or a method involving the use of radioactive materials for the deposition. However, we have been concerned here with the investigation of films of a type used for optical purposes and we believe that in this case it is the optical properties, rather than the actual thickness or density of the film which count.

It is hoped that the calculations and graphs in the theoretical part will be of use to other workers when attempting to determine the form of the deposit and may lead to applications not yet considered by the authors.

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Notes Added to the Proofs by the Authors.

The authors' attention has been drawn to an article entitled 'A Study of the Propagation Mode for Metallic Vapours in Shadow-Casting by Vacuum Evaporation of Au^{198} and Cr^{51}' by L. E. Preuss (*J. Appl. Phys.*, **24** (Nov. 1953), 1401-1409). This worker determined the vapour intensity distribution of conical baskets and cylindrical filaments using a radioactive evaporant. He found that the distribution of the deposit differed considerably from that based on point source emission. According to our results one would expect the intensity distribution of the cylindrical filament to approach that of an extended source but the amount of gold volatilised by Preuss from the spiral was insufficient to cover the heater uniformly, and thus the emission characteristics would not be comparable to that of a tungsten spiral completely wetted by the molten evaporant as used here. Preuss observed marked preferential evaporation from the open ends of both the basket and cylindrical filaments. He was concerned with the evaporation of minute quantities of metal (order of a few mg in weight) for shadow-casting purposes, whereas here we were concerned with the evaporation of large evaporant charges, as required for producing optical coatings, etc. For supporting minute charges in a spiral filament, the gaps between the turns must be made very small, and when an evaporant such as chromium is sublimated, only a small quantity of vapour is able to escape between the filament turns. Under these conditions, the vapour emission from a basket source approximates to that of a conical shell container. Inspection of the spatial distribution curves reported by Preuss show that they have marked similarity to the beaming effect obtained by P. Clausing (*Z. Physik*, **66**, (1930), 471) for free molecular flow from a short tube.

for References see Overleaf

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ALUMINIUM REFLECTING FILMS APPLIED TO GLASS AND PLASTICS

by Thermal Evaporation

S. Bateson, B.Sc., M.Sc., Ph.D. *Director of Research*
*Duplate Canada Ltd., Oshawa, Ontario, Canada**

PART I

Aluminium Films on Glass with Particular Reference to Front Surface Mirrors

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Summary

HEREWITH IS PRESENTED a study of the fundamental principles which govern the deposition of aluminium films upon various materials. The article is divided into two parts. Part I deals with films on glass supports and Part II with films on plastics.† The conclusions and notations are extended wherever possible to the commercial aspect of evaporation.

Sommaire

PRESENTATION D'UNE ETUDE des principes fondamentaux qui régissent la déposition de films d'aluminium sur des matériaux divers. L'article est divisé en deux parties. La première présenté ci-après traite de films sur support en verre et la deuxième de films sur plastics. Les conclusions et annotations sont étendues autant que possible aux aspects commerciaux de l'évaporation.

INTRODUCTION

THE SUBJECT MATTER of this paper is not new. Indeed, so many references exist that a comprehensive bibliography cannot be presented. Undoubtedly, much useful information still remains in the records of individual laboratories for whom such coatings have theoretical, technical or commercial value. In preparing this paper, therefore, the writer has attempted to present general principles and procedures which have proven themselves during some fifteen years experience; consequently, a good deal of information directly pertaining to the high vacuum process has been omitted in favour of process fundamentals.

Most people will agree that it is a simple matter to produce bright, durable aluminium films by means of a simple laboratory evaporator using a mercury pump and liquid air trap, for example. The literature abounds with description of this sort of thing, nevertheless, the steps involved in translating the process from an elementary to a commercial level are complex.

Probably the most outstanding property which strikes one upon examining the aluminium film on glass is its brilliance and tenacity of adhesion to the base. Strong bonding of this kind suggests that chemical action occurs between the film and the glass surface.

We shall begin by discussing the physical nature of the glass surface under atmospheric conditions and in so doing we draw upon the intensive work of Weyl[1].

THE GLASS SURFACE

The Structure

The structure of glass is governed by the principle

* MS. received March, 1953.

† To be published in the next issue (Vol. III No. 1)

that each Si^{4+} ion is surrounded tetrahedrally by four oxygen ions. Each oxygen is, on the average, shared by two Si ions.

When two fresh surfaces are created by fracturing a piece of glass, an equal number of units either with an uncoordinated oxygen or minus oxygen will remain on each side of the fracture. Using Weyl's notation these units make up a layer of structural units as follows[2]:

$$\left[Si^{4+}\ (\tfrac{1}{2}\ O^{2-})_3\ O^{2-}\right]^{-}_{\frac{x}{2}}$$

$$\left[Si^{4+}\ (\tfrac{1}{2}\ O^{2-})_3\right]^{+}_{\frac{x}{2}}$$

Although the layer is neutral, individual units are strongly polarised and will react with atmospheric water vapour:

$$\left[Si^{4+}(\tfrac{1}{2}\ O^{2-})_3\ O^{2-}\right]^{-}_{\frac{x}{2}} + \tfrac{x}{2}\,H_2O \rightarrow \left[Si^{4+}\ (\tfrac{1}{2}\ O^{2-})_3\ OH^{-}\right]^{\circ}_{x}$$

$$\left[Si^{4+}(\tfrac{1}{2}\ O^{2-})_3\right]^{+}_{\frac{x}{2}}$$

Presumably after exposure to the air, a freshly formed glass surface comprises a neutral layer of OH groups. It is suggested that bonding of a metallic layer to the glass is accomplished by chemical reaction with this layer.

A characteristic of the exposed OH groups is their ability to retain neutral water molecules by covalent hydrogen bonds. Consequently, if the glass is exposed to excess water vapour, a weakly bound layer will cover the surface:

$$\left[Si^{4+}\ (\tfrac{1}{2}\ O^{2-})_3\ OH^{-}\right](H_2O)_x$$

Not only will water be held by the polarised layer, but fatty acids as well.

The Cleaning

In practice, we are concerned primarily with glass surfaces which have been 'degreased' but which still retain a layer of water structurally bound to the exposed groups as we have described. This layer of bound water is not removed by ordinary chemical cleaning methods. Much more vigorous treatment is required such as sparking in air, cathode bombardment in a vacuum or heating of the glass *in vacuo* to at least 250°C.

Glass surfaces which have been exposed to these treatments are characterised by a 'black' breath figure which results from a lowering of the contact angle between the glass and water.

A study of the efficiency of various cleaning methods in removing residual films from glass surfaces preparatory to aluminising has been carried out by means described below.

Testing the Cleaning by Contact Angle Measurement

The contact angle between distilled water and glass was measured[3]. In practice, extreme variability of cleaning techniques had already been observed. Lack of precise information concerning the governing factors led to a quantitative study of contact angles and these were used as the criterion for various pre-treatments.

The methods of Bickerman[4] and Langmuir[5] were adopted with some refinements. Water droplets weighing on an average 3.3 mg. were placed on the glass surface by means of a 1 c.c. hypodermic syringe. Distilled water buffered to a pH of 7.08 ± 0.02 at 25°C. was used. During the tests room temperature was maintained between 70.0 ± 3.0°F. and the R.H. at $20.0 \pm 2.0\%$. A small correction to allow for evaporation was applied when computing very small angles. The contact angle may be measured directly, by an optical method[5] (see Fig. 1) or it can be calculated from the drop dimension[4]. Excellent agreement between both procedures was found.

In the first instance the glass was cleaned in the following manner :

(a) Careful swabbing of the glass surface with a detergent such as Orvus to which a mild

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abrasive such as precipitated chalk had been added.

(b) A tap water rinse followed by distilled water after which the glass was immersed in clean alcohol.

(c) Brisk buffing with tissue to effect the final drying.

Modifications of the method are possible but the procedure has not been bettered in its essence.

Then the contact angle was measured and its value used as a criterion, for the effectiveness of the foregoing treatment. Comparisons were made with a clean glass surface sparked in air by means of a Tesla Coil. The latter gave a 'black' breath figure[6] which has always been considered indicative of a really clean surface. Contact angles were measured after exposing the samples to room air for 5, 30 and 60 minutes. The results are summarised as follows :

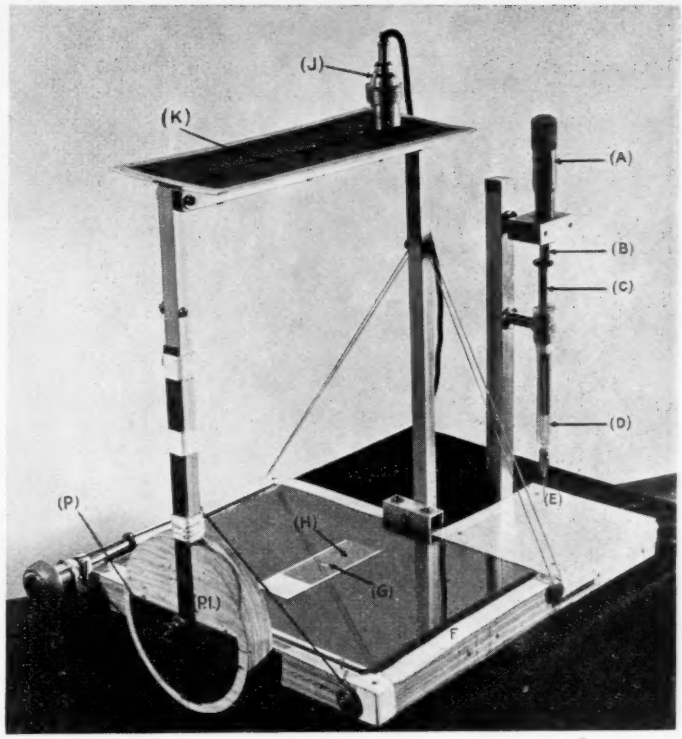

Fig. 1. Contact Angle Measurements of Glass Surfaces. Simple equipment suitable for measuring contact angles of less than 20°. (A) Micrometer thimble (calibrated in thousandths of an inch). (B) Micrometer spindle (depressing syringe plunger). (C) Syringe plunger. (D) Hypodermic syringe filled with distilled water of known pH. (E) No. 22 size s.s. needle. (F) Glass topped base. (G) Water drop on glass slide. (H) Microscope slide. (I) Light source on C/L of viewing slit. (K) Viewing slit (9.-cm. long × 5.0mm. wide). (P) Protractor (O° at top to 90° in the plane of the table). (P-1) Protractor arm attached to movable viewing slit.

Table 1

Treatment	*Contact Angles*		
	After 5 mins.	*After 30 mins.*	*After 60 mins.*
O/A/S (Orvus and Alcohol with sparking)	12.5° to 16°	17° to 20°	23° to 25°
O/A (Orvus and Alcohol only)	17° to 20°	21° to 23°	23.5° to 27°

Conditions of Test

Room Temperature - 70.0±0.3°F. Relative Humidity - 20.0±0.2% pH of Distilled Water - 7.08±0.02@25°C. Number of Tests - 6 each value

Three conclusions are fairly obvious :

(1) O/A cleaning does not yield as low an initial contact angle as O/A/S.

(2) After exposure to room air, glass treated by either method picks up contamination * until the condition of the surface is identical.

(3) The O/A/S treatment renders the surface particularly reactive to contamination. Contact angles as low as 9.5° have been measured by reading the angle as soon as possible after sparking.

Variations in the angle at different points on a clean glass surface are greater for O/A-treated glass than for O/A/S. Readings of the contact angle have an average deviation of ±5.0° for the O/A-treatment as compared ±2.0° for the O/A/S-treatment.

The foregoing observations illustrate the effectiveness of detergent-alcohol cleaning methods, and

* Layers of fatty acids, water vapour etc.

indicate that consistent results may be obtained provided reasonable care is taken. Moreover, the measurements show that any attempts to improve surface cleanliness still further by the use of acids or by sparking merely renders the surface highly 'reactive' to room contamination to which it must of necessity be exposed for some time before introduction into the vacuum system. Of course, it is tacitly assumed that grease and other gross impurities which might be present due to handling have previously been effectively dealt with by more vigorous methods if need be.

The foregoing characteristics are associated with preliminary cleaning and trouble may be minimised by the use of a proper technique in a controlled atmosphere. Nevertheless, even after the most careful preparation we have shown by contact angle measurements that the surface is still contaminated by a layer of water or fatty acid residues. It has been assumed that final removal of these is effected by cathode bombardment in the vacuum. Evidence for this assumption has been mainly empirical. Coatings applied to glass which have been exposed to a glow discharge show consistently better adhesion, less signs of deterioration and a 'blacker' mirror.

Efforts to substantiate these assumptions by contact angle measurements have yielded results which are somewhat confusing.

Microscope cover glasses were carefully cleaned with O/A; one set being arc-cleaned in addition to the pre-treatment. Half of each slide was covered with a second glass. The test pieces were pumped down in a bell-jar under various conditions. Control slides were prepared in the same manner and left in air for the same length of time. The contact angle readings after this treatment are given in the following Table II.

The following conclusions are drawn from these results.

(1) Films exposed to high tension cleaning during the rotary pump cycle and left in the bell jar until the pressure reached the flashing point showed just as much contamination as those which were not bombarded, see (*a*) and (*b*) in Table II.

(2) Films exposed to high tension cleaning during the rotary pump cycle and removed at once without opening the high vacuum valve were almost free from contamination. See (*c*).

(3) A control comparison is shown at (*d*).

The explanation is probably as follows :

Detergent ('Orvus') and alcohol cleaning followed by cathode bombardment in the vacuum produces clean but *reactive* surfaces. Subsequent exposure to the atmosphere within the system readily forms a secondary film over the clean glass surface as shown by the higher contact angles on the slides exposed in the bell jar. Present operating techniques allow the glass to become contaminated during the interval between bombardment and the flash point. Back-streaming from the oil diffusion pump during the time in which the poppet valve is open may largely account for this film formation. Therefore, it is most important to reduce to a minimum the time between opening the high vacuum valve and flashing.

Final Remarks on Structure

In this section, we have discussed how water molecules attach themselves to the highly reactive glass surface produced by cathode bombardment. Following Weyl's theory, various possibilities which exist at the glass surface exposed in the vacuum system have been summarised in Table III. The first case is not met with in practice. Moreover, the third condition may be modified by fatty acid radicals.

ADHESION OF THE FILMS

General Observations

One of the most striking characteristics of aluminium films is the tenacity with which they adhere to the substrate. As compared with gold, which easily rubs off, the difference is marked. Oxide formers such as magnesium, aluminium and chromium adhere firmly to the substrate while a noble metal such as gold does not. It is possible to list a number of metals in order of their adhesion to glass as shown in Table IV.

Adhesion between the metal and the substrate is

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Table II

Controls in Air				*Slides in Vacuum*			
Elapsed Time	*Treatment*	*Contact Angle*		*Elapsed Time*	*Treatment*	*Contact Angle*	
		Exposed Part	*Covered Part*			*Exposed Part*	*Covered Part*
Mins.		*Degr.*	*Degr.*	*Mins.*		*Degr.*	*Degr.*
25	O/A/S	25.0	11.0	25	(1) O/A/S (2) Pump to Flash Pt. No bombardment	27.2 (*a*)	15.7
20	O/A/S	19.1	10.2	20	(1) O/A/S (2) 15′ – Bombardment (3) Pump to Flash Pt.	27.2 (*b*)	14.2
25	O/A/S	21.5	14.8	25	(1) O/A/S (2) 15′ Bombardment and Release	14.2(*c*)	9.8
25	O/A	23.8	23.7	25	(1) O/A (2) Pump to Flash Pt. No bombardment	27.5	27.8
25	O/A	24.9	24.4	25	(1) O/A (2) 15′ – Bombardment (3) Pump to Flash Pt.	23.0	22.7
25	O/A	23.2	21.0	25	(1) O/A (2) 15′ Bombardment and Release	14.7	25.0(*d*)

Conditions of Test: Room Temperature 70 ± 2°F. pH - 6.92 ± .02 R.H - 35 ± 3%. Pressure at bombardment approx. 50 micron. O/A/S - Detergent ('Orvus'), Alcohol, sparked.

Table III

Surface Conditions	*Formula of Surface Units*	*Structural Formula*
Freshly broken :—	$[Si^{4+}\ O^{2-}(\frac{1}{2}\ O^{2-})_3]^-$ $[Si^{4+}\quad (\frac{1}{2}\ O^{2-})_3]^+$	O O O | | | Si Si Si Si Si Si
After atmospheric exposure :—	$[Si^{4+}\ (\frac{1}{2}\ O^{2-})_2\ 2OH^-](H_2O)_2$	$H_3O\ H_3O$ $H_3O\ H_3O$ $H_3O\ H_3O$ | | | | | | O O O O O O V V V Si Si Si
After cathode bombardment sparking or heating :—	$[Si^{4+}\ (\frac{1}{2}\ O^{2-})_2\ 2OH^-]^\circ$	6 H_2O ↑ OH OH OH OH OH OH V V V Si Si Si

Table IV

Ion	*Crystal Radius (Cation)*	*Radius Ratio (Cation/ Anion)*	*Coordination Number*	*Electro Negativity*	*Ion*	*Crystal Radius (Caution)*	*Radius Ratio (Caution/ Anion)*	*Coordination Number*	*Electro Negativity*
Si^{4+}	0.65	0.37	4	1.8	Ag^+	1.26	0.72	8	—
Al^{3+}	0.72	0.41	4 or 6	1.5	Au^+	1.37	0.78	9	—
Cr^{6+}	0.81	0.46	4 or 6	—	O^-	1.76	—	—	3.5

greater for cations of lower ionic radius, and for those with coordination numbers similar to Si^{4+}. Silver and gold do not belong to this group.

We have already described the ionic layer at the surface of the substrate, and we have seen that under ideal conditions the glass surface at the instant of flashing should be in a highly ' reactive ' state due to the exposed OH groups. Nevertheless, such a condition may not easily be realised due to contamination from the vacuum system as shown. Aluminium is extremely active, hence it is to be expected that the vapour at the glass surface will react chemically with these OH centres, displacing hydrogen.

The Effect of Oxygen

Experimental evidence has been presented by Stahl[7] that even in well degassed high vacuum systems there are present quantities of oxygen. Electron diffraction ring patterns of the oxides Al, Mg, Mo, Ni, etc., appear at film thicknesses of 1 micron or less. This oxygen must be released by the gettering action of the metallic vapour and must come from residual adsorbed gases in the system. At a constant rate of evaporation, the space immediately surrounding the source becomes depleted in oxygen until equilibrium is reached. Consequently, as the evaporated layer builds up on the substrate, its character changes gradually from the oxide to the metallic lattice. If, for example, evaporation is interrupted, a second oxide interlayer is formed etc. The intermediate oxide layer between the glass and the metal will grow thicker until the amount of metal deposited is increasing too fast for the oxygen supply.

Stahl estimates a limiting thickness of 1 micron for the oxide-bonding layer.

Structure of the Oxide Layer Formed

We infer from the foregoing that the metallic layer is bonded to the glass surface by an intermediate oxide layer.

Alumina exists in a number of crystalline forms as well as an amorphous one.

α - Al_2O_3 and γ - Al_2O_3 have the following co-ordination numbers for O^{2-} ions :

Ion	*Crystalline*		*Glass*
Al^{3+}	$\alpha - Al_2O_3$	6	4
	$\gamma - Al_2O_3$	4 or 6	

Therefore, both form tetrahedra in glass and could compose the oxide layer at the surface.

γ - Al_2O_3 is hygroscopic, a fact which might account for the formation of ' matt ' surfaces due to a transition of the bonding layer in the presence of humidity.

α - Al_2O_3 on the other hand is stable. Both are associated with tetrahedral and octahedral co-ordination. These crystal structures occur in alumino-silicates and are possible forms of the oxide. That the oxide layer is amorphous is an additional possibility.

We have already shown that a layer of ' bound ' water may be attached to the glass. If this is not removed before the aluminium is evaporated, a secondary reaction may continue between the metal and excess water, with the release of hydrogen. This would explain the bubbling which often occurs in poor films. The action is local and proceeds to an end point. Further evidence is required to clear up these questions.

OPTICAL PROPERTIES OF ALUMINIUM FILMS

Properties of the Ideally Prepared Film

Of all the metals with useful optical properties, silver and aluminium stand out pre-eminently because of their excellent reflectivities. Their properties are complementary in several respects. Aluminium can fill certain applications where silver would fall down. On the other hand, silver, with proper protection, still has a wider use as a second surface mirror for the visible spectrum. Nevertheless, since we are limiting our discussion to first surface mirrors, we can safely make the following statements.

Aluminium films have a higher reflectivity than newly deposited silver for all wavelengths shorter than 4,100 Å. For ultra-violet work down to 3,000 Å,

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aluminium has a reflectivity greater than 80% and is, therefore, very useful for interferometer mirrors in this region. At 2,000 Å, the reflectivity falls very sharply to about 50%. In the visible region of the spectrum vacuum evaporated or chemically deposited silver is superior to aluminium. Because it tarnishes rapidly, however, one must apply some form of protective coating. This procedure lowers the reflectivity and does not completely eliminate tarnishing over a period of time. The reflectivity of aluminium films between 4,000 Å and 8,000 Å should be about 90%. Above 8,000 Å the reflectivity rises steadily throughout the infra-red reaching 97% at 12 microns.

Operational Conditions Affecting Quality

The quantitative results quoted above are descriptive of an ideally prepared aluminium film. It has long been noticed that wide variations in reflectivity occur under different conditions of deposition. Three important factors have a bearing upon the brightness of aluminised mirrors. These are :

(1) Degree of vacuum.
(2) Presence of contamination in the vacuum system.
(3) Rate of deposition.

Vacuum. The first condition should, with modern pumping equipment, present no problem, and provided a pressure of not more than 0.1 micron Hg is maintained, the films should show no signs of 'brown'.

Contamination. Contamination within a high vacuum system may, indirectly produce films of low reflectivity. For example, if water vapour is present to any extent in the system, the pressure rise which accompanies gettering by the aluminium vapour interposes a buffer or scattering layer of gas between the source and the working surface. The effect is to produce films with a brownish or bluish cast. Likewise, oil vapour and other forms of contamination can affect the brightness of an aluminium mirror by direct deposition on the glass. In actual practice, we have found these two conditions of the greatest importance in their bearing on quality.

Deposition Rate. The effect of the rate of deposition upon the reflectivity of evaporated films has been extensively studied[8] by means of the electron microscope. In general, it may be stated that metallic films formed by rapid evaporation consist of small crystallites; those deposited by slow evaporation show a much larger amorphous area. This behaviour may be explained by the mobility of the metallic atoms on the substrate. In the case of gold and silver, the effect is pronounced; however, the reflectivity of aluminium films seems less affected by the rate of deposition, at least over the range met with in practice. For very low rates of evaporation of the order of 20 minutes to 20 hours, it has been shown that for a semi-transparent film, the loss in reflectivity could amount to as much as 20% at 4,000 Å[8]. Since we are dealing with evaporation times of less than one minute, it is not likely that this could happen; however, aluminium films deposited even in one minute are not quite as bright as those produced by rapid evaporation of the metal in a few seconds. Our own experience inclines us to believe that this is due to contamination from the de-gassing of the system.

The Need for Surface Protection

Freshly evaporated aluminium films are extremely soft. After they have stood for a short time, an oxide layer is built up which increases their durability. In order to obtain really durable first-surface mirrors, some form of protective layer must be applied. This can be accomplished in several ways:

(1) A layer of aluminium oxide may be formed by chemical action or by anodising.
(2) Films of silicon monoxide[9], silica, or magnesium fluoride may be evaporated on to the fresh aluminium surface.

No matter what technique is used, a compromise must be reached between abrasion resistance and objectionable colour saturation. For chemically hardened and anodised surfaces, a protective layer is formed at the expense of the metal, hence thicker metallic deposits must be laid down. The thickness of the protecting coat may be carefully controlled right up through the various orders of interference until the metal is completely oxidised. The thick-

ness of evaporated protective layers cannot be controlled to the same degree; however, it is feasible to deposit silica in thicknesses below the first order of interference and still obtain sufficient protection. Silicon monoxide films are extremely durable, but they must be carefully controlled if intense coloration is to be avoided.

ALUMINISING ON AN INDUSTRIAL SCALE—PRODUCTION OF FRONT SURFACE MIRRORS

Several years ago, the writer had occasion to review the development of aluminising[10] particularly the commercial aspect. During the Second World War, mass production techniques were introduced and great strides were made particularly in vacuum pumps and equipment. Since the War a good deal of this information has been published and particular attention has been paid to methods of protecting the aluminium film.

The discussion following below applies to production on a large scale and the figures quoted represent normal process averages.

Preparation of Glass Surfaces

A number of well-tried methods for cleaning glass surfaces previous to aluminising are well known. Evidence has already been presented (*loc. cit.*) for choosing one of the simplest procedures.

The difficulty of cleaning glass in humid weather is well known. Furthermore, the presence of water vapour in the vacuum system has a pronounced effect on the aluminium film. Our own experience proves that atmospheric control in the vacuum room and at the final glass ' buffing station ' as well, is necessary if steady production is to be maintained. In support of this, figures are presented correlating both total rejects of aluminised mirrors with day to day changes in humidity (see Fig. 2). Although rejections for all causes are included, the dependence of those defects upon moisture is obvious.

Humidity is expressed in grains per cubic foot. Atmospheric control prevented large temperature fluctuations but was not able to hold the humidity at a low value from day to day. This was later remedied by installing adequate air conditioning.

Aside from making the glass surface harder to clean, water vapour affects the process in another way. The pumping speed is reduced partly due to contamination of the forepump and diffusion pump oil and partly because of the added load on the pumps.

Atmospheric humidity affects the pumping cycle in still another way. The residual water vapour reacts with aluminium in the vapour phase during firing. The hydrogen which is displaced

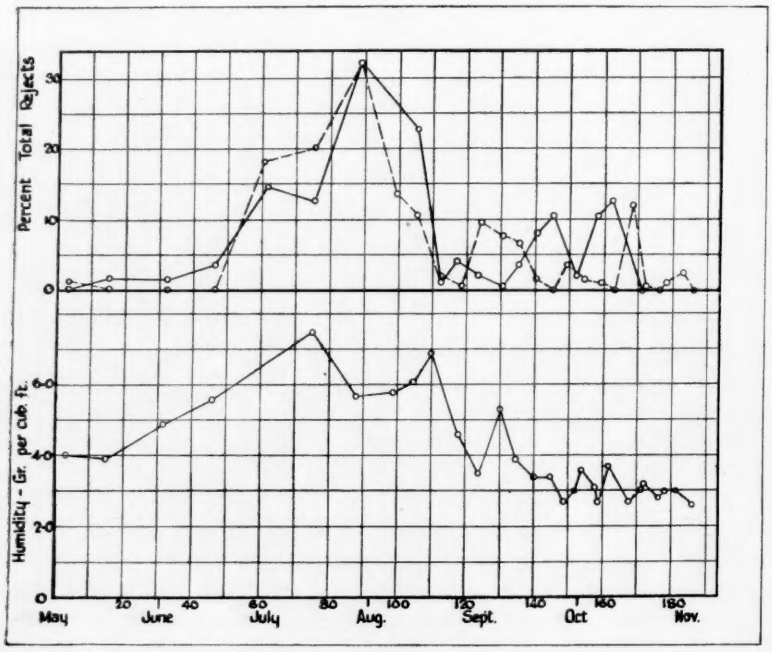


Fig. 2. Correlation Chart. The chart compares total rejects for all causes with humidity conditions. The dotted and solid lines refer to two different mirror sizes.

must be swept out of the system by the pumps. If their capacity is unequal to the task, a pressure rise will take place instead of the usual fall due to 'gettering'.

Rating System for Quality Control of the Finished Front Surface Mirror

The defects which are common to aluminium films on glass divide into distinct groups. By considering those associated with the coating itself, faults due to scratches, sleeks, short finish and so forth, are eliminated because they are properties of the glass base.

The aluminising process comprises four distinct operations :

(a) Preliminary glass cleaning.
(b) Pre-conditioning in the vacuum.
(c) Volatilisation of the aluminium.
(d) Deposition of the protective layer.

It is possible to establish ten types of defect, each of which has a characteristic appearance under the inspection light and is sufficiently unique to differentiate it from any other. Each of these peculiarities is a direct result of fault at one of the four stages to which reference has been made.

The practical evaluation of a commercially acceptable aluminium front surface mirror involves a range of attributes, the overall rating of which determines the quality of the mirror. In order to arrive at a reproducible set of standards, the rating system we devised had to satisfy the conditions postulated below :—

1. Successful Adaptation of Quality Control Procedures to the Production of Front Surface Mirrors.
   (a) Choice of attributes to facilitate evaluation.
2. The Development of a Rating Chart and a proper basis for it.
   (a) Breakdown of overall quality into a number of attributes which shall be, as far as possible, independent of each other.
   (b) Use of the system (1) as an acceptance test for the mirrors and (2) as a check on the process.
3. Eye-rating *versus* Quantitative Laboratory Tests.
   (a) A test is of value only if it affords useful information.
   (b) Agreement of scoring in visual testing.
4. Determination of a Final Standard of Acceptance based on
   (a) customer requirements,
   (b) cost and
   (c) practicability of the process.

Controlling the standard of front surface mirrors produced on a commercial basis presents a number of unusual difficulties. In the first place, it is not possible to apply a single quantitative measurement which will pass or reject a mirror; moreover, sampling methods must be adopted when the output is large. To make matters worse, each mirror is judged by the customer probably as much on appearance as by performance. Therefore, any inspection to which

Fig. 3. Typical inspection booth suitable for front surface mirror rating. The lower lamp fastened to the table is the inspection light proper.

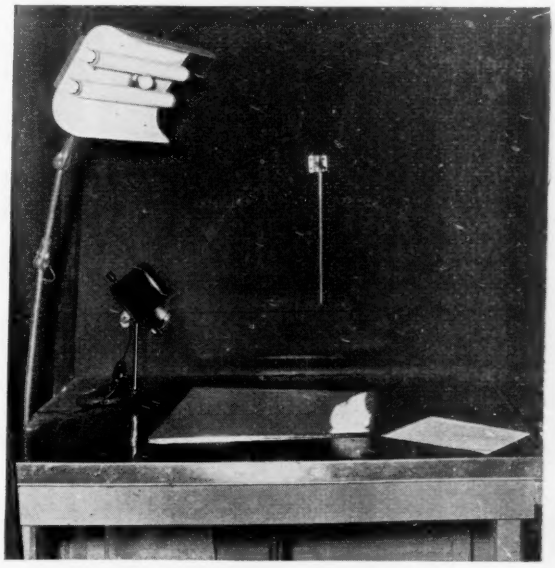
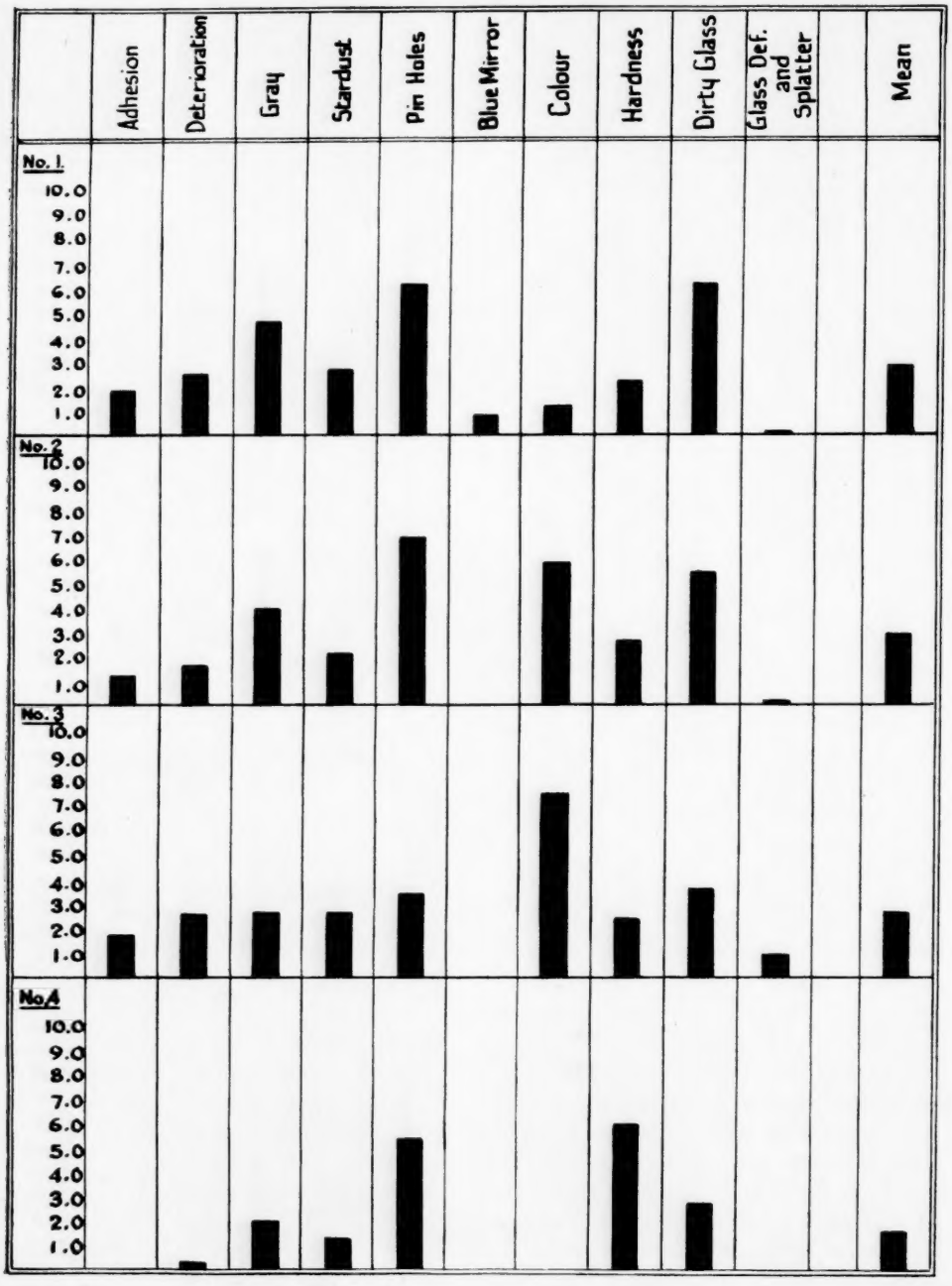


Fig. 4. Front Surface Mirror Rating System. Mirrors 1, 2 and 3, were a random sample from the same origin. Mirror 4 was of another origin and obviously rates the best. (Least score on the chart.)



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the mirror is subjected must be based on appearance as well as performance. The problem therefore, resolves itself into determining which attributes are pertinent and how they shall be weighted.

The purpose of the rating chart is to break down quality into a number of attributes each of which shall be, as far as possible, independent of the other. Actually, as mentioned before there are ten of these. The method of inspection, their appearance under the inspection lamp and the method by which they are scored is discussed below in detail (see Fig. 3).

The rating system was designed not only as an acceptance test for quality control of the mirrors, but also to check the various steps which enter into the process.

The scoring is based essentially on eye-rating under controlled inspection conditions. Providing each attribute is clearly defined and the scoring adjusted to cover only the variation which is distinguishable and no more, the system becomes a powerful semi-quantitative method of analysis. Agreement between independent observers using the rating system is remarkably good.

Final acceptance standards based on this system, will, of necessity, be determined by customer requirements, commercial limitations in the process and cost. Some thought must be given to the tolerances which are mutually acceptable to the customer and manufacturer. Obviously, there exists an overall score which a mirror must not exceed in order to pass. On the other hand, a single attribute, if its score exceeds a prescribed limit, could be a cause for rejection, although the overall rating was within the acceptable limit. These matters are provided for by the special design of the chart.

Inspection Procedure

Whenever possible, twenty mirrors of approximately the same manufacturing period were selected and examined under the rays of the Burton Fresnel lamp set at oblique incidence, but with the mirror field rendered dark by means of a black cloth covered table. The inspection was carried out quickly and an overall judgment made on a large surface area without stopping too long to consider every detailed flaw.

Catalogue of Defects (See Fig. 4)

The ten defects are defined briefly as follows, remembering that in the scoring system adopted, 10 points against a mirror for a particular defect represent maximum imperfection :

Adhesion. Scotch tape pressed across the mirror (or diagonally) and stripped off. Complete let-go of the metal constitutes maximum score. Varying degrees of failure are observable, however, by holding the mirror against the light and noting the increase in frequency and size of pinholing along the path of the tape.

Deterioration. A pattern of bright specks usually near the edge but in extreme cases over larger areas of the mirror. The specks are actually tiny blisters in the metal which catch the light. At its worst, deterioration may even resemble a matt surface.

'Gray'. A background covering the whole surface of the mirror with a uniform 'scum'.

Star Dust. Similar to deterioration in appearance but not patterned, usually covering large areas but with wider separation between bright specks. Due to short finish in the glass.

Pin Holes. When the mirror is held up to the light, innumerable holes of different sizes may be seen. In a freshly coated mirror their number is small, but after brushing the surface, pin holes show up.

Blue Mirror. Showing a bluish background against the light.

Colour of Protective Layer. The colour was first evaluated by holding the mirror perpendicular to white paper for overall hue. Lack of uniformity in the coating was next checked by observing the mirror at grazing incidence.

Hardness. Fifty circular rubs are made with a wad of cotton wool using considerable pressure. The scratch pattern is then examined under the lamp. Gross scratches are discounted as arising probably from abrasive caught up in the cotton. A background of hairlines usually shows with sufficient clarity to judge the degree of hardness by their denseness.

Dirty Glass. Dirt on the glass looks much like the gray background in general appearance but is characterised by streaks and patches, even finger marks, following the path of the buffing cloth.

Glass Defects and Splatter. Chips, scratches, and crush marks are the usual glass defects. Splatter refers to lumps of aluminium (or SiO) deposited on the reflecting surface. Distance from the edges of the glass, of course, should be taken into account in weighing this kind of fault.

Part II dealing with Aluminium Films Applied to Plastics will be published in the next issue.

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TRAINING REPORTS

VACUUM COURSES AT THE ROYAL TECHNICAL COLLEGE, GLASGOW*

Introduction

AN ACCOUNT OF COURSES in Vacuum Technology at the Northampton Polytechnic has already been given by F. Y. Poynton. The courses at the Royal Technical College, Glasgow, have only been running since 1950 and have tended to develop along somewhat different lines. In order that the reasons for this may be fully appreciated, it is necessary to digress somewhat, and discuss the background against which the course has grown.

Associateship Course in Applied Physics

A new course in Applied Physics started in Glasgow in October 1949, and has now been running for four years as a full-time day course. It is a four-year course for the Associateship of the Royal Technical College, and is designed to train physicists for industry by giving a thorough grounding in mathematics and classified physics together with practical training in the use of industrial equipment. In order to obtain a pass with honours, the students have to complete an investigation and write a thesis during their fourth year. The final qualification is officially recognised in many quarters as equivalent to an honours degree.

The course in Vacuum Physics was planned as an integral part of this course, and also as a suitable field for many research projects by students and post-graduate workers. The practical work in Vacuum Physics forms the background to a course on the kinetic theory of gases and the basic theory is developed in conjunction with, and illustrated by, the practical work, and acquires a reality which is frequently absent from the average course on kinetic theory. In the later part of the course dealing with applications of vacuum processes, the properties of thin films used as anti-reflection coatings and in interference filters are studied and form an extension of the theory of interference and interferometers covered in the applied optics section of the course.

Each section of the Applied Physics course has been similarly chosen with an eye to the theory which it would illustrate and accompany, the basic idea being that fundamental physics should be taught as far as possible in close association with its applications. On this count alone a course in Vacuum Physics is justified, but it also precedes further sections dealing with X-ray crystallography and electron microscopy. The knowledge and experience of vacuum equipment already acquired enable the students at this later stage to focus their attention on the basic theory of the processes and to handle the instruments intelligently without being unduly distracted by the subsidiary vacuum equipment and accessories. Electron dynamics and wave mechanics accompany the course on electron microscopy, and the X-ray crystallography forms an extension of earlier work, the foundation in atomic physics having been laid in the early part of the course.

Further details of this course and the general background have been given in an article by Professor J. S. Rankin, *Bull. Inst. Phys.*, April 1953.

Evening Courses

Evening courses were started in 1950, partly to meet the demand from industrial concerns and partly to suit students who wished to take a Higher National Certificate in Applied Physics, but courses for the Higher National Certificate are now becoming available in other local colleges and the course is being modified to post-graduate standard suitable for endorsement on Higher National Certificate. This is in accordance with the general policy of the College, to expand post-graduate work and supplement the effort of neighbouring institutions by providing facilities beyond their resources, rather than to compete in the same field. In view of the type of student and limited time available a certain amount of knowledge has always been assumed in this course, which thus lacks, to some extent, the carefully integrated character of the day course. As the course acquires its post-graduate character, more knowledge of basic principles can naturally be assumed, and more advanced work will be undertaken. This course runs for approximately 10 weeks, with 1 lecture period and 1 practical period each week, and is devoted mainly to measurements and practical applications. Time is limited, and the laboratory work consists mainly of standard methods of gauge calibration, measurement of pumping speeds, and a study of the effects of gases other than air, with later exercises on leak detection and vacuum deposition by sputtering and evapora-

* M.S. received July, 1953

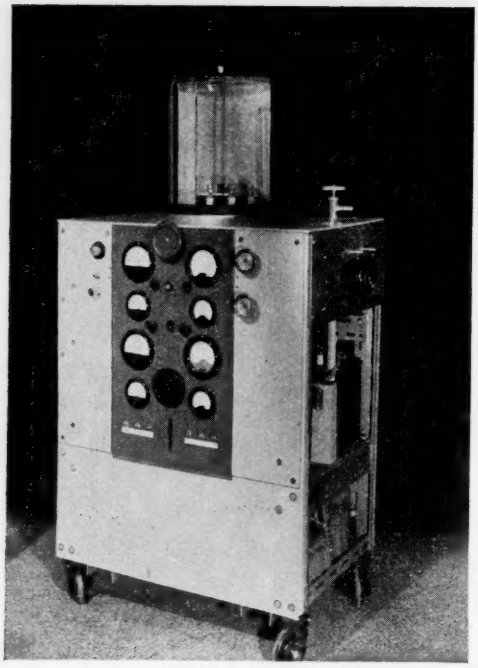

Fig. 1. Standard vacuum unit as used in the laboratory.

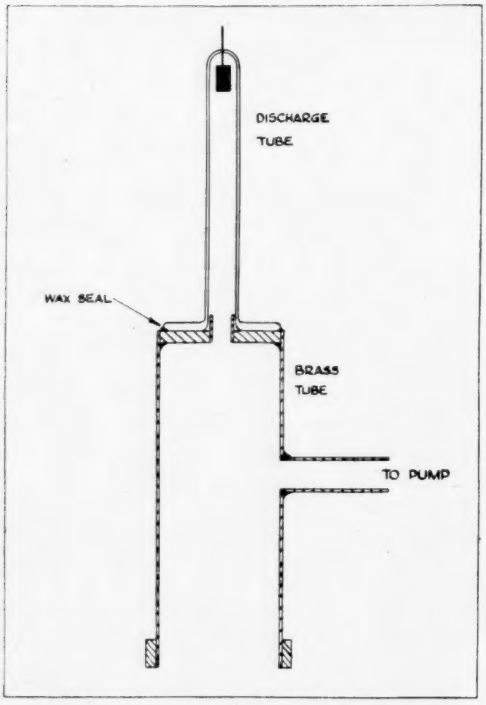

Fig. 2. Leak testing device for connecting plugs inserted in the baseplate of the vacuum unit.

tion. In contrast to the London courses, valve production is somewhat neglected since there is little demand for it in Scotland, and alternative courses on the electronic side already exist. More time is devoted to optical, chemical and metallurgical applications.

Our experience of the type of student attending this course agrees with that of the Northampton Polytechnic, and our records show scientists from government and industrial laboratories, post-graduate students and technicians from the University and various firms, either using or manufacturing vacuum equipment.

Accommodation

One room was available for electron microscopy and vacuum physics, the choice being dictated by the provisions required for the microscope that it might be free from interference and vibration. The room chosen was well away from heavy engineering machinery and had existing concrete pillars rising directly from the foundations. These were used to support the electron microscope to minimise vibration. An adjoining darkroom made the position more suitable.

The room was partitioned to separate the electron microscope and its associated equipment from the Vacuum Physics section. Additional water supplies were laid on and wall benches were installed, but without extensive structural alterations it was not possible to provide drainage along more than one wall. Arrangements were made to provide six working positions along this wall. Benches along the other walls provided space for glass-blowing, a mercury still, acid cleaning baths, sputtering equipment, auxiliary optical measuring equipment and experiments which do not require a water supply.

Equipment

In view of the shortage of working positions at which a water supply is available, the general plan has been to provide portable pumping units which can be wheeled into any suitable position as required. The units have been constructed on rectangular frameworks of Dexion angle, and the floor area occupied by each is only 15 inch. × 24 inch. which allows economical use of the available space.

The units are approximately 3 ft. 6 inch. high, and

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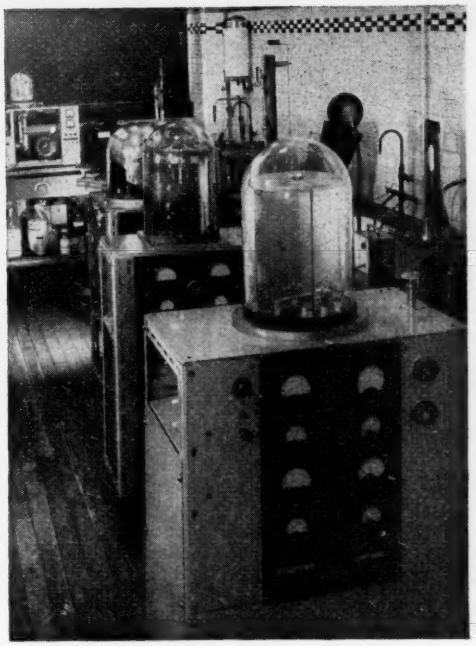

Fig. 3. View of one corner of the High Vacuum laboratory showing some units in their working positions, and a miniature evaporation unit in the background.

each unit has a bell jar as working chamber, with a high vacuum or flap valve and diffusion pump mounted beneath the baseplate. A rotary pump on anti-vibration mountings occupies the bottom of the unit. A metal P_2O_5 trap is mounted on top of the rotary pump and the main pumping line runs from the trap to a vacuum stopcock on top of the unit, with side-connections through valves to the backing side of the diffusion pump and directly into the working chamber. The stopcock comes just above bench level and is used with auxiliary gauges, etc., when the unit is being used only as a rotary pump. The tubing is almost entirely metal and 'Yorkshire' plumbers fittings which have been recessed to take O-rings where required are used so that the system is easily assembled and dismantled. The pumping arrangement is thus conventional, but by choice of different types and makes of pumps, each unit presents a different combination, and the student is introduced to as wide a range as possible of modern types. Metal oil diffusion pumps are generally used, but one or two mercury pumps are available and serve a useful purpose in demonstrating the necessity for liquid air traps.

The baseplate is machined with holes counterbored on the underside to suit metal plugs as commonly used in the trade. These plugs can be tightened down on to an O-ring seal by a nut on the top side of the baseplate, and are used to connect gauges, leak valves, electrodes, etc., directly to the working chamber. All-glass gauge heads are generally used and are waxed into these metal plugs to allow easy insertion and removal. During leak testing, the individual plugs are easily tested by placing over each in turn, the small metal hood shown in Fig. 2. This is connected by a rubber tube to the stopcock on top of the unit and sits on a rubber ring placed round the plug to be tested. A Tesla coil applied to the discharge tube indicates the degree of vacuum and the test can readily be made more sensitive by closing the stopcock after evacuating the tester and observing any change in the discharge whilst isolated from the pump. Even a small leak into such a small volume is readily detected.

Calibrated gauges are undesirable for teaching

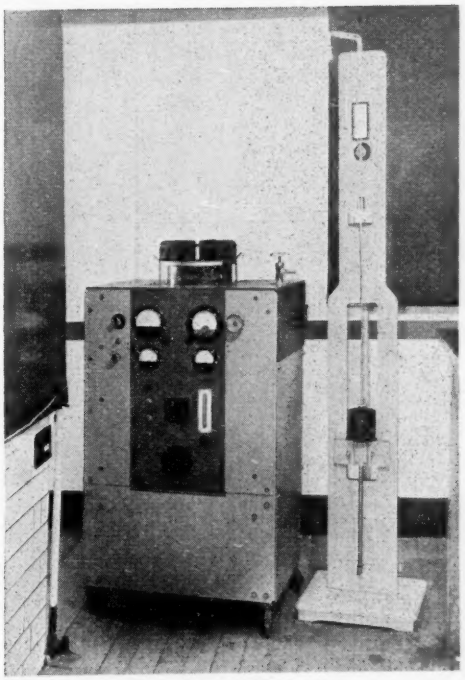

Fig. 4. Sputtering unit showing vacuum chamber and air-cooled cathode. The rubberless McLeod gauge serves to indicate the scale.

purposes and glass gauge heads are preferred so that the student can see clearly the construction of the gauge he is using. Each unit is normally fitted with a Pirani gauge and a hot filament ionisation gauge and the circuits are kept as simple as possible. The Pirani gauge is operated directly from an accumulator and the ionisation gauge is fed from a simple power pack contained in the unit. The necessary meters are mounted on the front panel, which hinges forward to give easy access to the underside of the baseplate. The gauges are thus built into the unit but the student has to adjust voltages and currents for each reading in the same way as would be necessary with a gauge head and independent power supply.

The only other equipment contained in each unit is a high voltage power supply controlled by a Variac with alternative a.c. or d.c. output. The output is connected to two insulated terminals on a side panel for external use, and can also be connected to electrodes through the baseplate. This power pack is used only in more advanced experiments, and can be used to produce a cleaning discharge or for sputtering.

When in use, the units normally stand sideways to the wall with the short side against one of the wall benches. In the initial stages the units are used for pumping only, and auxiliary equipment—discharge tubes, McLeod gauges, etc.—is connected to the stopcock, which comes just above bench level. The student gradually becomes familiar with the unit and obtains practice in connecting equipment by butted joints, cone joints, etc., whilst using only the rotary pump. As the units come into full use, the task of instruction is made easier by the common design and the fact that all students can work on the same experiment. This may appear extravagant, but actually the cost of all the gauges supplied is, because of their simple form, less than the cost of one or two manufactured gauges and the risk of breakage is far less serious. By the time separate experiments are issued, the students have been working with the standard unit for several weeks and are familiar with its operation.

Each working position has three 5 A plugs, and one 15 A plug, and the student is supplied with a rubberless McLeod gauge on a stand in addition to a vacuum unit. A liquid air trap is attached to the stand, and the outlet comes almost level with the top of the unit. It can either be connected directly into the working chamber by a short tube passing through one of the plugs in the baseplate and bent to project at the back of the unit, or joined to the stopcock on top of the unit. A wide-necked Thermos flask in a plastic container is used for liquid air and brought up under the trap. The plastic container minimises student risk due to flask breakages, and is more sturdy than an unprotected flask.

This McLeod gauge is the only calibrated gauge supplied. Short McLeod gauges are also used but have a linear scale and are uncalibrated. Other calibrated gauges are available as required for advanced work, but the general practice has been to purchase only one calibrated gauge of each type to be available for demonstration and use as required. In the later stages of the courses the units are set up and used for evaporation and sputtering.

In addition to these general purpose teaching units, a miniature evaporation unit of commercial design is available and a special sputtering unit has been constructed. This uses a moulded glass cylinder to form the working chamber and a large air-cooled cathode sits on top of the glass cylinder. The cathode is earthed and the baseplate is insulated and maintained at high voltage. Surfaces up to 8 inch. diameter can be coated and operation over long periods is possible without overheating. The small volume of the working chamber gives an extremely short pumping time even with the small two-stage pump which is used. The General Electric Co. Ltd. have kindly given us permission to use this arrangement, which is a feature of one of their patents.

Practical Work

The range of experiments covered includes measurement of pumping speeds, gauge calibration, use of discharge tube as a vacuum gauge, measurement of flow through tubes, leak testing, evaporation and sputtering, and follows the same lines as the Northampton syllabus which has already been given. The practical work is kept in step with the lectures, since all students can have the same experiment. This has two advantages: Firstly, that a student does not have to attempt an experiment before covering the theory in his lectures, and secondly, that time is saved in explanations and supervision in a course where time is extremely valuable.

Teaching aspects have been emphasised, but the design of the vacuum units was necessarily influenced by the requirements of research. Nevertheless, we consider the design finally adopted to be a fair compromise, for teaching purposes, between the demands of economy in time spent in setting up experiments and of enabling students to assemble their own equipment. In the early stages of the course, when the units are used for pumping only, the students are required to fit up the pumping systems for themselves, and familiarity with vacuum systems is gradually acquired before the students make use of the complete unit.

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Research

Two types of research work had to be considered. Students following the day course for the Associateship of the Royal Technical College have to write a thesis in their fourth year on an investigation. Obviously the problems tackled have to be clearly defined and preferably complete in themselves, so that the work can be completed in the limited time available. On the other hand, post-graduate work for a Ph.D. degree can embrace a wider field.

Research at the present time is mainly concerned with the properties of thin films, both metallic and dielectric. The electron microscope used as a diffraction unit is a valuable tool in many investigations, and there is close collaboration between the two sections of the department. Tolansky techniques are used in measuring film thicknesses, and the thickness of films is frequently monitored during deposition, using optical methods. With dielectric films, interference colours can be observed by direct reflection from the front surface, or interference bands can be produced by creating the necessary conditions for multiple-beam interference. The interference bands are normally viewed by reflection from the rear, and a reversion spectroscope has been found valuable for accurate measurements. The diffuse edges of the bands make accurate use of an ordinary spectroscope difficult, but direct vision spectroscopes are frequently used because readings can be taken without any adjustments and the lower accuracy is offset by speed of reading. On metallic films, transmission and reflectivity measurements are made using photo-electric methods where necessary, and the effects of deposition methods and of substrates are being studied.

In view of the importance of optical methods, space has been set aside and most measurements are made in the Vacuum Physics laboratory. Additional equipment and facilities are available in the Applied Optics section when required.

Conclusions

The existing ten weeks evening course has been found to be very cramped and the possibility of a longer course or an intensive full-time course has been considered, but development in this direction depends upon industrial demand.

It is hoped that this account of our methods, and the development of our courses in Vacuum Physics will be of interest to other colleges and industrial users of vacuum equipment.

C. WEAVER.

LECTURES ON INDUSTRIAL APPLICATIONS OF HIGH VACUUM TECHNOLOGY

As in previous years Northampton Polytechnic arranged for a course of 12 lectures under the above heading during last years session (February 24th to May 26th, 1953) in addition to the established course on vacuum technique which is a permanent feature of its educational programme. Although a number of new topics were introduced the attendance at this special course was somewhat below expectations (between 30 and 50) and for this reason no arrangements have been made to repeat the course in the current session (1953/54).

As far as we know the substance of these lectures has not been published elsewhere but our readers may wish to be acquainted with their content. Therefore, we print below a summary of each lecture. In a few cases additional information is given on points covered by the original paper.

The Production and Measurement of Ultra High Vacua.

Presented by : *G. H. Metson.*

The diffusion pump and chemical 'getter' have made the production of ultra high vacua (say 10^{-10} mm. Hg) a matter of relative simplicity. Such a vacuum has, however, little virtue in itself; it is only valuable as environment to the working of some useful machine. It is the actual working process of such machines that give rise to vacuum problems—usually in the form of gas generation leading to destruction of the machine itself. The art of the vacuum tube engineer centres therefore around two problems : Firstly, the production of a high initial vacuum and secondly, the more difficult task of preventing excessive gas generation during working life.

The principal gas generators in modern high vacuum tubes are set out in a rough order of prevalence :

(*a*) Thermal outgassing of metal components.
(*b*) Thermal outgassing of glass and mica components.
(*c*) Chemical breakdown of contaminating metal films under electron bombardment.
(*d*) Chemical breakdown of glass under random electron bombardment.

To these sources might be added the probability of slow generation of electrolytic oxygen from the alumina used as heater insulant.

The getter is well able to deal with these gas

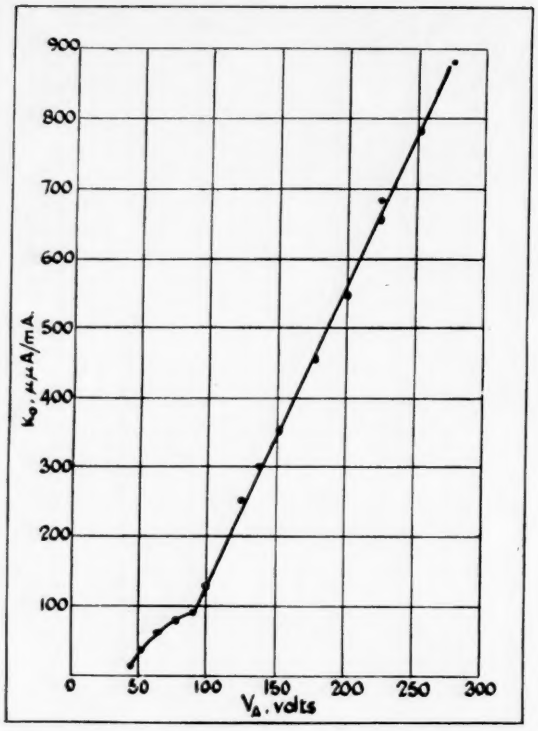

Fig. 1. Example of a ko/VA characteristic. (G. H. Metson).

generations so far as quantities are concerned but unfortunately the gases tend to find their way preferential to the cathode under ionic influences. Protection of the delicate cathode from positive ion bombardment is probably the single most important reason for producing ultra high vacua.

Measurement of ultra high vacua is at present based almost exclusively on the common triode ionisation gauge working in the range 100-200 volts whose probability of ionisation is roughly constant. Unfortunately the gauge generates soft X-rays at its anode and these rays, falling on the positive ion collector, eject photo-electrons which are measured in the external circuit as an equivalent positive ion flow. This X-ray effect sets a limit to the sensitivity of the gauge. A typical characteristic of the photo-electron flow is shown in Fig. 1, and is seen to be voltage-dependent in a characteristic manner. The interfering effect of the X-rays can clearly be mitigated up to a point by reducing the geometrical surface area of the ion collector or by enclosing the collector in a retarding electric field sufficiently strong to deny the emergence of the photo-electrons.

Table I. Minimum detectable probe gas pressure change (J. H. Leck).

Detecting Instrument		*Approximate* $\triangle P$ *(Microns)*
Mass Spectrometer	(Helium)	10^{-8}
,, ,,	(Coal Gas)	10^{-7}
Ionisation Gauge	(Butane)	10^{-7}
,, ,,	(Carbon Dioxide)	10^{-6}
Pirani Gauge	(Butane)	10^{-3}
,, ,,	(Hydrogen)	2×10^{-3}

Leak Detection

Presented by: *J. H. Leck*

In most modern methods of leak detection the position of the leak is determined by detecting the presence inside the vessel (whilst the vacuum pumps are operating) of some probe gas or vapour applied externally.[1] Usually either a mass spectrometer, an ionisation gauge or a Pirani gauge is used as the probe indicator. For the ionisation and Pirani gauges butane (calor gas) is the most effective probe gas. Where butane is inadmissible, because of the danger of explosion, carbon dioxide and hydrogen are the best gases available for the ionisation and Pirani gauge respectively. For the mass spectrometer helium is ideal, but prohibitively expensive in this country. Methane (the CH_3^+ ion) is no more than a factor of 10 worse than helium and is therefore satisfactory in almost all cases.

The minimum detectable leak in any system is $\triangle P/S$, where $\triangle P$ is the minimum change in probe gas pressure which can be detected and S is the pumping speed at the detector. Values of $\triangle P$ are given in Table 1 in order that a comparison of the different methods may be made.

As the minimum detectable leak $\triangle L$ depends upon S, the factor $\triangle P$ rather than $\triangle L$ is the better basis on which to compare the different methods. The sensitivity for the ionisation and Pirani gauges will be less than the calculated values when the total leak rate is large (gas pressure at the gauge greater than 10^{-4} and 10^{-1} microns respectively) because the fluctuations in air pressure will increase $\triangle p$.

[1] The halogen leak detector described by W. C. White, and J. S. Hickey, in *Electronics*, **21**, March 1948, 100, is a notable exception.

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Surface Coatings on Optical Components

Presented by : *W. Zehden*

The presence of thin metallic and non-metallic films on the surface of an optical component affects the reflecting and transmitting properties of this surface. By suitable choice of material and thickness of a film or films, it is possible

(1) to produce high reflectivity and stop the transmission (mirrors),
(2) to produce a required ratio of transmission and reflection for white light (neutral filters and beam splitters) or high transmission-low reflection for one wavelength range, and low transmission-high reflection for another wavelength range ('dichroic' beam splitters and interference filters),
(3) to reduce the surface reflection, and, in doing so, to reduce stray light and increase transmission (blooming).

In general, these films are deposited *in vacuo* by means of evaporation or sputtering. As their thickness is only of the order of a wavelength or fraction of a wavelength of visible light, they exactly reproduce the shape and structure of the surface on which they are deposited, *e.g.*, if the object is a highly polished optical flat, the coated surface will remain highly polished and flat.

The thickness of metallic films is controlled during deposition either visually by comparing their transmission with standards, or the transmission is measured by means of a photocell. Non-metallic films are usually controlled by observing their colours in reflection, either visually by comparing them with colours of standard coating, or with a photocell by illuminating with monochromatic light and observing the appearance of reflection maxima and minima. There are, of course, other methods used in many individual cases.

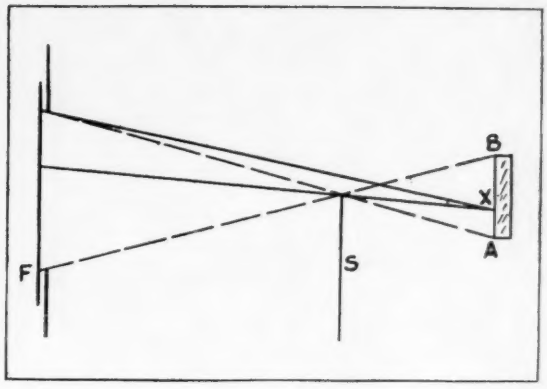

Fig. 1. Arrangement for the evaporation of rhodium for the manufacture of neutral wedges.

As a special item, a technique for making a neutral wedge was described. With rhodium as the evaporated material, it is possible to make a linear density wedge beginning with zero density and going up to the maximum density required, without using any moving parts in the vacuum chamber. Fig. 1 shows the arrangement. F is the filament, a rhodium plated closely wound tungsten spiral, the ends of which are shielded so that the exposed part can be taken as uniformly heated. A-B is the glass to be coated, and S is a stop. At any point X of the glass AB, the length of filament seen during evaporation is proportional to the distance A-X. The thickness of the coating obtained will, therefore, go up linearly

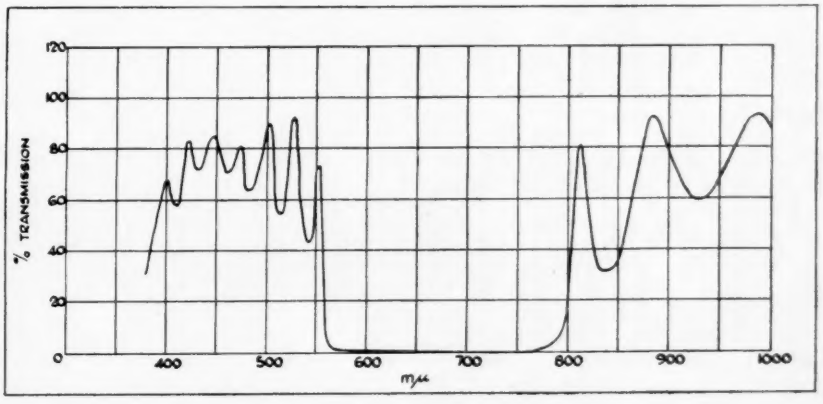

Fig. 2. Typical transmission curve of a 19 layer coating (zinc sulphide alternating with cryolite). Optical Thickness of each layer ¼ wavelength at 685 mμ.

from zero at A to maximum value at B. The latter is controlled during evaporation in the usual way with light source and photocell.

As an example of the performance of a non-metallic multilayer system, Fig. 2 shows a typical transmission curve of a 19-layer coating. This particular coating makes a very good minus-red filter. It should be noted that, with this type of coating, practically all light which is not transmitted is reflected.

Large Accelerator Vacuum Systems

Presented by : *L. Riddiford*

During the last twenty years particle accelerators have been developed from pieces of scientific equipment of not unconventional size and complexity to the stage where a very great amount of engineering and technical effort is required for any installation which is to bear comparison with those already in existence in a rapidly changing field. In the case of the large cyclic accelerators in particular, in which the atomic particles are made to travel very great distances in the process of acceleration, vacua as good as those normally employed in a laboratory 'pumping-bench' system are required in systems having volumes of as many as tens of thousands of litres. For this reason advances in high vacuum technology have to a large extent gone hand in hand with accelerator development.

The degree of vacuum required is dependent on the aperture available for the vacuum tube, the injection energy, the rate at which the particles are accelerated, and the radius of the machine which determines the distance they travel. The materials used and method and details of construction of the vacuum tank or tube are to a large extent restricted by other aspects of the accelerator design, which makes difficult the maintenance of those high standards that are necessary in sound vacuum technique. That the standards have been maintained by technical developments is apparent. Many large synchro-cyclotrons and electron-synchrotrons, which require pressures not worse than 1×10^{-5}, and the Brookhaven proton-synchrotron requiring a similar pressure, are now in operation at particle energies of from 100 to 2,000 MeV. In the case of the 1,000 MeV Birmingham proton-synchrotron, the vacuum tube is now complete and the pressure less than the required one of 1×10^{-6} mm. Hg.

The Birmingham synchrotron uses a hollow porcelain vacuum tube constructed of sixty pieces each of interior cross section about 4 inch. $\times$ 18 inch. sealed together by rubber-to-metal bonded gaskets to form a circle 32 feet in diameter. It is evacuated by five 16-inch oil diffusion pumps each operating through a large stainless steel manifold. The Brookhaven synchrotron uses a tube consisting of sheets of non-porous rubber stretched over a ribbed stainless steel structure which provides the necessary mechanical strength. The tube, of cross section 6 inch. $\times$ 48 inch. is constructed in four quadrants each about 50 feet long, and is evacuated by twelve 20 inch diffusion pumps. A still larger proton-synchrotron under construction at Berkeley uses a tube formed of quadrants about 100 feet long.

As a result of experience with the Brookhaven accelerator, and recently conceived ideas on synchrotron design, the accelerators of the future will use very extended tubes of small cross section, evacuated by a larger number of small diffusion pumps. Machines currently under consideration at Brookhaven and the European Nuclear Physics Laboratory would use a 2-inch. diameter tube with a radius of 300 feet. Because of the strong focussing of the gas scattering electrons with the new principle the pressure required will be only the same as in existing machines for the same injection energy.

Vacuum Evaporation for Industrial Processes

Presented by : *L. Holland*

Two main aspects of vacuum evaporation were considered *viz.*, (i) general deposition techniques, (ii) the coating of volatile substances. Both topics were the subject of considerable research in the lecturer's laboratory.

It was shown that the flat strip heaters used for vaporising dielectrics and metals had a vapour intensity emission corresponding to that of a plane surface and not, as often described, point source emission.[1] The similarity between the emission of molecular radiation and that of radiant energy from a diffuse reflecting surface has been shown by Knudsen.[2] The thickness, t, of a film deposited on a surface at a distance, r, from a small plane emitting source is given by :

$$t = \frac{M \cos\phi_1 \cos\phi_2}{\rho \quad \pi \quad r^2}$$

where ϕ_1 and ϕ_2 are the angles of vapour incidence at the source and receiving surface, M the mass

evaporated and ρ the evaporant density. Evaporated coatings of volatile metals such as Zn and Cd do not follow the distribution law because at high evaporation rates vapour atoms are not rectilinearly propagated.

Volatile metals cannot always be condensed to form thin films unless either the deposition rate is very high or the condensing surface is seeded with a thin film of metal of low volatility, *e.g.*, silver, copper, etc. The seeding technique was used during early work on molecular rays, see Frazer[3], and applied industrially for condensing Zn on to capacitor tissues.

There is a need for evaporating alloy films for resistors and beam splitters, etc. but the general subject of alloy evaporation had been neglected. Dushman[4] had shown that the partial vapour pressures P_A and P_B of the components A and B in a binary alloy could be estimated from Raoult's law by treating each component as a solvent, *i.e.* $P_A = X_A P_1$ and $P_B = X_B P_2$ where P_1 and P_2 are the vapour pressures of the pure metals and X_A and X_B their mol concentrations. The method must, however, be used with caution since Raoult's law, normally, only holds for high concentration of a volatile component. The evaporation rate E of a substance *in vacuo* is proportional to $P\sqrt{M}$, where P and M are the vapour pressure and molecular weight respectively. By combining the foregoing formulae one obtains an approximate ratio of the evaporation rates of the components in a binary alloy in terms of the vapour pressure of the pure components, *i.e.*

$$\frac{E_A}{E_B} = \frac{X_A P_1 \sqrt{M_A}}{X_B P_2 \sqrt{M_B}} = \frac{W_A P_1 \sqrt{M_B}}{W_B P_2 \sqrt{M_A}}$$

where W_A and W_B are the weight concentrations. For simultaneous evaporation of the components in a binary alloy E_A/E_B must equal W_A/W_B which requires that $P_1\sqrt{M_B}/P_2\sqrt{M_A}$ equals unity. A table of the ratio $P/\sqrt{M}$ had been compiled for a number of pure metals and this suggested that binary alloys based on either of the two groups (i) Al,Cr,Sn,Cu and (ii) Fe,Au,Ti,Ni might evaporate at normal rates without undue fractional distillation of the constituents. Such alloys can be termed 'constant evaporation rate' mixtures and should not require a specialised evaporation technique for producing homogeneous coatings. Alloys of Sn:Cu and Al:Cu have been found to evaporate without undue component separation.

Many factors influence the evaporation characteristics of an alloy other than that of vapour pressure. For example, a binary alloy may tend to form a two-layer film when slowly evaporated from a filament heater and a single homogeneous layer when rapidly volatilised. At high evaporation rates the filament temperature is very much greater than that of the evaporant when this is in the form of a molten droplet. Molten alloy flows from the droplet over the filament surface where it is 'flash' evaporated.

In the vacuum coating of volatile substances such as plastic components it was shown that the volatiles are desorbed by diffusion of the liquid within the porous solid and evaporation at the plastic surface. Some plastics, such as nylon and cellulose acetate, shrink in volume as their moisture content falls and their diffusion coefficient decreases accordingly. Degassing can be accelerated by raising the plastics temperature, but care is required to avoid undue plasticiser evaporation from plasticised materials, and by designing the component to have a minimum of diffusion path. Released volatiles may contaminate the pumping system, *e.g.* plasticisers of intermediate vapour pressure ($\simeq$ 1 micron Hg vapour pressure) may be condensed in the diffusion pumps, and the more volatile water vapour condensed under compression in the rotary pump. Oil stripping systems are therefore essential on both rotary and diffusion pumps. In one method[5] of purifying rotary pump oils the affected oil is allowed to flow into a number of trays arranged in cascade and air continuously bubbled through the oil.

[1] HOLLAND, L. and NEWMAN, N. J., *Rev. Sci. Instrum.*, **23**, (Nov. 1952), 642.
[2] KNUDSEN, M., *Kinetic Theory of Gases*, (Methuen & Co. Ltd., London, 1946).
[3] FRAZER, R. G. J., *Molecular Rays* (University Press, Cambridge, 1931).
[4] DUSHMAN, S., *Scientific Foundations of Vacuum Techniques*, (John Wiley & Sons Inc., New York, 1949), 764.
[5] Subject of a provisional patent application.

Freeze Drying

Presented by : *L. G. Beckett*

The vacuum engineering problems and some theoretical problems associated with the design and construction of freeze drying plant were outlined. The need was emphasised for considered alterations to the requirements of the biologist for readily sterilized and cleaned apparatus which, though automatic as far as possible, must be of the simplest construction and lay-out.

It has been assumed, in a general way, that for satisfactory preservation all materials must be dried to a very low final moisture content. In some countries specifications have been drawn up which cover the maximum permissible residual moisture content. These specify a moisture content below

0.5% by dry weight of the product. Undoubtedly there are many substances which show better keeping qualities with increased dryness. On the other hand there is now evidence that it is possible that some biological materials can be spoilt by overdrying. Suspensions of bacteria fall into this category. Methods must be employed, therefore, which limit the final residual moisture content of the material. Hitherto this has been achieved biochemically by including a polysaccharide in the suspending medium, in which the organisms are dried. This method is somewhat haphazard and a more precise method of obtaining a predetermined residual moisture content has been evolved. In the suggested method the dried product is exposed to a system containing water vapour at a predetermined saturation pressure until its pressure equilibrates with the latter.

Research work on these lines will be helped by a recently devised vacuum procedure of determining residual moisture content by means of equilibrium vapour pressure.

Glass-to-Metal Seals for High Vacuum Apparatus. Presented by : *O. Adams.*

Although much has been written and many theories proposed to explain the mechanism by which vitreous enamels adhere to iron, comparatively little has been published on the allied problem of the adherence of glass to metal.

Glass-to-metal seals are usually made in air at high temperatures and thus the metal oxidises during the process unless precautions are taken to prevent this. Consequently glass is separated from the metal by a layer of oxide and most glass-to-metal joints have a characteristic colour due to the reaction of the oxide layer and the glass.

This oxidation of the metal would seem to be necessary for the formation of a good seal between glass and metal since A. W. Hull and E. E. Burger[1] have stated that molten glass will not 'wet' the surface of clean metal in an atmosphere of hydrogen, pure nitrogen or carbon dioxide. The glass did spread rapidly over the surface however if the metal was oxidised.

However it has been possible to make alkali-free glasses adhere to tungsten by heating in an atmosphere of dry hydrogen. The resulting seals appear to be free from any oxide but it has not yet been possible to prove that this is so.

J. H. Partridge[2] has stated that the surface of iron became slightly oxidised when heated in the presence of molten enamel in carbon dioxide or nitrogen at low pressure and that a slight visible oxide film was sufficient to promote adhesion.

Metal oxides are quite soluble in glass at seal-making temperatures and thus adherence between metal oxide and glass is usually very good. Mechanical strength tests on seals have shown that fractures occur either in the glass near the joint or at the interface between metal and metal oxide, with oxide adhering to the glass. Fracture seldom occurs in the oxide layer itself unless the layer is too thick.

This the nature of the metal oxide and its adherence to the parent metal are of great importance in glass-to-metal seals.

For example, the oxide layer on 50/50 nickel iron alloy is mainly ferric oxide with a phase of the magnetite type containing nickel. During the manufacture of a glass-to-metal seal, however, the oxide appears to change and the only oxide detectable in the final seal is magnetite. There is a considerable expansion difference between magnetite and the original alloy so that adherence between the two is poor.

For this reason, it is usual for nickel iron to be plated with copper before being sealed into glass. The copper is then coated with a thin film of fused borax (as is usual with copper-glass seals) to prevent excessive oxidation during seal manufacture.

The greenish oxide layer on 26% chromium iron, however, appears to consist of a mixture of chromic and ferric oxides and the thermal expansion of such a mixture (between 8 and 10×10^{-6}) per °C depending on the proportions) is not greatly different from that of the alloy itself or the glass which is sealed to it. Glass adheres firmly to alloys of this type.

These are but two examples of one aspect of the problem of adhesion between glass and metal and much more information is required before the mechanism of this adhesion can be satisfactorily explained.

[1] *Physics*, **5**, 1934, 384-405.
[2] PARTRIDGE, J. H., *Glass-to-Metal Seals*, (The Society of Glass Technology, Sheffield, 1949).

Vacuum Technology in the Valve and Cathode Ray Tube Industry. Presented by : *R. O. Jenkins.*

The subject of vacuum technology in the valve and cathode ray tube industry is covered in three lectures. The first lecture introduces the subject and then deals

with the properties of the various materials used in valves and cathode ray tubes and their pre-treatment. In particular the properties of pure tungsten, thoriated tungsten and oxide coated cathodes are discussed and how these properties affect the vacuum techniques that can be used during processing valves. Vacuum and hydrogen cooking of components and getters of the dispersed and non-dispersed types are described. The vacuum properties of hard and soft glasses and treatments usually employed are also detailed.

The second lecture covers the evacuation of transmitting valves and cathode ray tubes. Suitable vacuum systems are described and processing schedules for transmitting valves from the high power type tungsten cathode down to low power oxide cathode types are discussed, and the general principles on which they are based. Treatment of valves with various gas fillings is also covered. The processing of various sizes of cathode ray tubes is dealt with similarly.

In the third lecture, vacuum systems for bench pumping of receiving valves are described, together with typical evacuation schedules.

The extension of this to mass-production systems and vacuum schedules is finally outlined.

Editors Note. The summaries of the papers have been compiled by the lecturers themselves and have been received between July and December 1953.

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Personal Data of Authors

ADAMS, O., Research Laboratories, The General Electric Co. Ltd., Wembley, Middlesex.

BECKETT, L. G., Research Laboratories, W. Edwards & Co. (London) Ltd., Manor Royal, Crawley, Sussex.

HOLLAND, L., Research Laboratories, W. Edwards & Co. (London) Ltd., Manor Royal, Crawley, Sussex.

JENKINS, R. O., Research Laboratories, The General Electric Co. Ltd., Wembley, Middlesex.

LECK, J. H., Dept. Electrical Engineering, University of Liverpool, Brownlow Street, Liverpool, 3.

METSON, G., Research Branch, Post Office Engineering Dept., Dollis Hill, London, N.W.1.

RIDDIFORD, L., Physics Dept., The University, Edgbaston, Birmingham 15.

ZEHDEN, W., Hilger Div., Hilger & Watts Ltd., 98, St. Pancras Way, Camden Road, London, N.W.1.

LETTERS TO THE EDITOR

An Investigation of a Metal Knudsen Manometer

Sommaire

Brève description d'une jauge de Knudsen métallique. Les détails du procédé d'étallonnage comprenant les pressions entre 10^{-3} et 10^{-4} mm. Hg sont donnés. A la fin de la lettre des effets du dégasage sont discutés.

IN A PREVIOUS paper W. Steckelmacher[1] has described and investigated a metal Knudsen manometer of his own construction. A similar instrument has been worked with in this laboratory.

This gauge is constructed in mainly the same way as Steckelmacher's although there are certain differences. Thus the vane is suspended in a single-ended tungsten wire, the necessary damping being achieved by means of two or three permanent magnets. The heaters consist of two helical resistance wires of a temperature running at about 30°C.

The connection to the vacuum system is made by a standard taper joint at the top of the envelope. The reading device is of the common lamp-and-scale type, and pressure readings may be taken by means of calibration curves belonging to the instrument. It has two ranges, 10^{-3}–10^{-4} and 10^{-4}–10^{-5}mm.Hg, controllable by a switch on the auxiliary transformer.

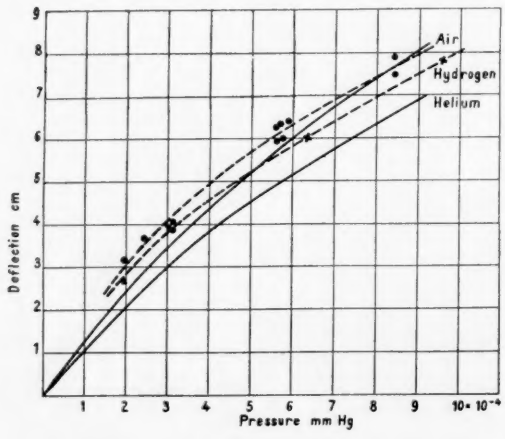

Fig. 1. Calibration curve for dry air and hydrogen.

The present work included a calibration of the gauge in connection with a general survey of its properties. The calibration was made by means of a pressure-reducing device similar to that used by Knudsen in his fundamental works[2]. It consisted of a number of glass bulbs with known volumes, joined together by a tube fitted with stopcocks or mercury cut-offs, which at one end was connected to a barometer and at the other to a mercury diffusion pump and liquid air trap. In this way the Knudsen gauge could be filled with gas up to a pressure which could be computed by Boyle's law. Before the Knudsen gauge was mounted in the system it was made sure by comparison with a precise McLeod manometer that the pressure obtained did agree with that computed by Boyle's law. This was found, in fact, to be the case throughout the range considered here. Our reason for performing the calibration in this way instead of in the one most commonly used was, that we wanted a calibration as absolute as possible without any auxiliary device.

The results of the calibration for dry air and hydrogen are shown on the attached Graph (Fig. 1.) Solid curves (for air and helium respectively) are due to a calibration made in the usual way, the dotted ones (for air and hydrogen) refer to this report. It should be noticed, however, that on account of a breakage the suspension wire had to be shortened by about 10% before the measurements were made, so the results should be expected to be correspondingly too low.

The calibration of the low range 10^{-5} mm.Hg proved to be impracticable in the present way because of a most disturbing gas development, which must be ascribed partly to the internal metal surfaces and partly to a possible minute leak. From our results we realize that the pressure in the gauge after cutting it off for reading will increase remarkable during the reading. In fact, the rate of increase was found to be on average 1×10^{-5} mm.Hg per minute.

As pointed out in Steckelmacher's article metal gauges are designed for use in large metal systems where gassing defects are overcome by increasing the pumping speed. In the light of this it is not surprising, that working with the gauge under these circumstances will meet with difficulties. The example merely shows what will happen when doing so. However, this does not prevent metal gauges from giving correct results when properly used. We consider it

Fig. 1. Back-streaming of fluid from an 02 oil diffusion pump through a straight pipe.

essential that it has a very wide bore connection just as in Steckelmacher's mentioned above, as correct pressure measurements in metal systems depend upon the fact that the pressure gradient in the connecting tubes is negligibly small.

This work has been performed at the Royal Technical College, Copenhagen, under supervision of Professor Dr. R. E. H. Rasmüssen, to whom I would convey my best thanks for steady encouragement and words of instruction.

H. G. JENSEN.

Royal Veterinary and Agricultural College,
Copenhagen, Denmark.

27th April, 1953.

[1] STECKELMACHER, W., *Vacuum*, **1**, (Oct. 1951), 266.
[2] KNUDSEN, M., *Ann. Phys. Lpz.*, **444**, (1914), 581.

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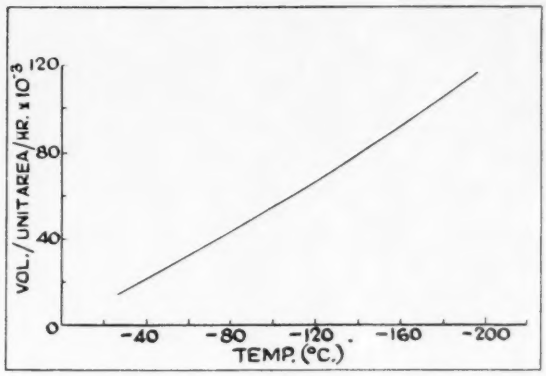
On the Assessment of Back-Streaming in Vacuum Systems

Sommaire

L'auteur décrit une méthode pour essayer l'éfficacité de pièges refrigérés dans des systèmes à vide. La chose est particulierement important quand, dans un appareil à metalliser sous vide par example, la pièce est maintenue à une température inférieure à l'enceinte. En vue de se rendre compte de la quantité d'huile rétro-évaporée de la pompe, et pouvant contaminer la surface à recouvrir, une cuve d'Adam a été employée, contenant de l'eau dont la surface a été soupoudrée de talc pulvérulent très fin. Une aire connue de la surface à recouvrir est trempée dans l'eau et l'huile se repend pour former un film monomoléculaire à travers la surface. L'huile repousse le talk et la surface qui en est recouvert est définie par des arrêtes très nettes. A l'aide de moyens additionels de calibrage la quantité d'huile est déterminée. Les résultats de l'enquête sont représentés par des graphiques.

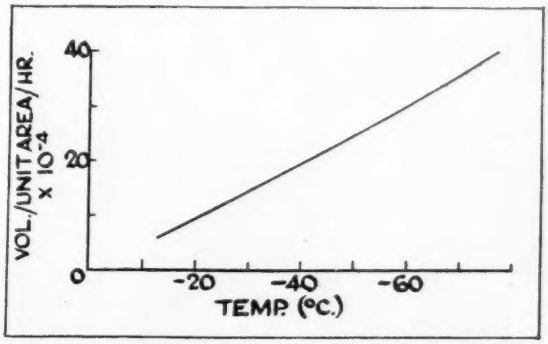
THE DANGERS which arise with diffusion pumps because the molecules of the boiling fluid diffuse or 'back-stream' into the evaporation chamber have been realised for some time[1] and various arrangements of baffles are commonly used to overcome this

Fig. 2. Back-streaming of oil from an 02 oil diffusion pump through a double right angled bent pipe.



difficulty[2]. The problem may be particularly serious if it is necessary to maintain a substrate *in vacuo* at a temperature significantly lower than that of the surroundings. In such cases the use of cooled traps is obvious, but means of checking the efficacy of such safeguards is desirable. The purpose of this note is to report a method used in studying the behaviour of oil diffusion pumps which is simple and easy to apply.

The very small quantity of oil which is deposited on a substrate can be measured using an Adam trough. A uniform layer of a fine powder is sprinkled over a clean water surface in the trough[3]. A known area of the substrate is dipped into the water and the oil spreads to form a monomolecular film across the surface. The oil pushes back the powder and the area of the film is defined by a sharp boundary. If two parallel glass barriers, waxed to prevent wetting, are arranged some distance apart and the oil deposited between them, then the area of the resulting rectangular film can be readily measured. The most satisfactory powder in our experience is fine unscented talc.* It is important not to use too much powder. If it is desired to convert the areas of the



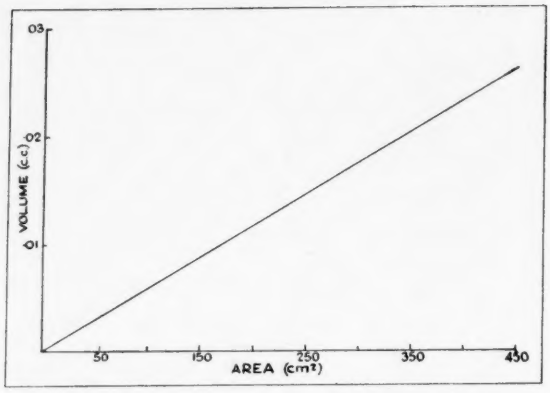


Fig. 3. Calibration graph of volume and surface area.

films into volumes, a calibration is easily performed with the aid of a miniature pipette.

The results of two experiments are shown in Fig. 1 and 2. The pumping system was an O2 Oil diffusion pump charged with Apiezon A oil and backed by a rotary pump. The calibration curve is shown in Fig. 3, the slope of which indicates that the thickness of the oil layer was 0.62 microns. In the first experiment the diffusion pump was connected to the evaporation chamber by a straight tube with no taps and Fig. 1 shows the volume of oil condensed per unit area per hour as a function of temperature. In the second case, Fig. 2, a double right-angled bend was inserted between the pump and the baseplate. The effect was clearly to reduce the amount of oil deposited to about one tenth of that with the straight line.

The method is obviously simple, quick and flexible. The accuracy obtained is of the order 20%. This might without difficulty be significantly improved, but the primary purpose of such experiments is to obtain an assessment rather than an accurate estimate of the amount of oil which is likely to contaminate any particular specimen.

S. G. BRADLEY,
G. K. T. CONN,
G. K. EATON.

Department of Physics,
The University,
Sheffield, England.

22nd July, 1953.

* We are indebted to Dr. W. D. Oliphant of Messrs. Ferranti who drew our attention to this and to Messrs. Ponds who supplied the powder.

[1] STRONG, J., *Modern Physical Laboratory Practice*, (Blackie & Son, Ltd., London, 1940), 116.

[2] GUTHRIE, A., and WAKERLING, R. K., *Vacuum Equipment and Techniques*, (McGraw-Hill Book Co. Inc., New York, 1949), 89.

[3] ADAM, N. K., *The Physics and Chemistry of Surfaces*, (Oxford University Press, Oxford, 1930), 35.

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BOOK REVIEWS

High Vacuum In Industry. Post-Graduate Study by M. GROSSMAN, ET AL. (Manufacturing course of Professor Georges Doriot at the Harvard Graduate School of Business Administration, 1953). Copyright by Marvin Grossman, et al. Pp. 64.

The report was written by seven graduates under the auspices of the Harvard Graduate School of Business Administration and shows a remarkable grasp of the subject as a whole. The sources of information quoted are very reliable and extensive and the report gives a comprehensive survey of the future of industrial vacuum technique in America. Whether the same future is possible in this country is open to doubt. The annual turnover for the whole American industry is quoted as $20,000,000 and 70 percent of this business is handled by four large vacuum equipment companies, each ploughing back a good proportion of its profits into research and development funds—: 'National Research Corporation is spending over 5% of every sales dollar on pure product research . . . .' This seems to be the standard practice for avoiding excess profits tax and appears to be encouraged by the Government who also sponsor a large number of research projects, particularly those associated with atomic energy. The American public seems to think that

the future of the vacuum industry is very sound, as Consolidated Vacuum had no difficulty in raising $3,000,000 by public subscription for the specific purpose of buying Distillation Products in 1952. N.R.C. in the same year easily raised $1,500,000 for increased research and manufacturing operations. These figures are on a much larger scale than the present expenditure in this country.

The future developments predicted by the report for the vacuum industry fall into two classes:—

(a) the improvement and reduction in price of existing equipment as competition between the four major companies increases, and

(b) the application of vacuum technique to new and existing processes.

In the latter group it is pointed out that serious competition can be expected from well-established techniques and unless the vacuum method achieves either a considerable reduction in cost or a vastly superior product, it is not likely to be adopted except in special cases.

The report is written in several sections, the first is about the industry as a whole and the others each apply to some section of the industry in detail. Each section has a short introduction for the benefit of the non-technical reader which adequately describes the basic principles involved in each process and then deals at length with the various applications. Running costs are frequently given in detail to enable them to be compared with the costs of other existing techniques.

The section on basic equipment is intended primarily to describe all the components likely to be found in a complete vacuum plant. The only new developments of note are the extensive use of silicone oils in diffusion pumps and the use of gas ballast rotary pumps which enable water vapour to be pumped without the necessity of using drying agents.

The metallurgical industry is stated to hold the brightest future because there are frequently no alternatives to the vacuum process. Metals, previously unobtainable, have been produced and the Government has given considerable financial help to this project. Refining, purification, melting and casting, heat treating and the coating of metals with a heat-resisting coating of another metal are all discussed.

A new group of materials called Cermets or Ceramels are mentioned and these consist of ceramics bonded by metals. They are produced by a sintering process and have been used as turbine blades.

The vacuum coating industry has a section of its own but is frequently mentioned throughout the report, for example, in the electronic section is mentioned the production of standard components, including triodes and printed circuits. Prices are not competitive but quality and smallness are the selling points here. The authors of the report feel that development will be extremely slow in this field.

Vacuum technique excels in the production of mirrors and the coating of optical lenses with non-reflecting films. Work has now begun on the large-scale coating of ophthalmic lenses, T.V. screens, large glass sheets, etc. The use of metal-coated plastics for decorative and practical purposes is well developed, and although these processes are used in this country, the American manufacturer seems to be more aware of their possibilities, for example, the motor car industry is using metal-coated plastic components instead of chromium-plated metal on the outside of cars.

Great scope exists for sheet metal protected by an evaporated coating instead of by plating but a successful continuous evaporating source has not been developed. Another desirable material is metal-coated plastic sheet which is a superior product to metal-foil laminate and which would be used in large quantities in the packaging industry if it were readily available. None of these materials can be produced in large enough quantities at the moment to make the process attractive economically.

The production of drugs and other heat-sensitive chemicals by vacuum distillation and the drying of various antibiotics and vaccines is discussed at length and here again there is considered to be room for expansion.

Vacuum drying also enters into the section on the food industry, which is entertaining as well as informative. The great difficulty here is to persuade the public to accept common foods in new forms and with unaccustomed flavours. Various anomalies exist, for instance vacuum-dried orange juice when reconstituted is stated to have a superior flavour to fresh juice, and although coffee loses its aroma, it is still a successful product because it is easily soluble. Apple juice, on the other hand, has met with only moderate success because of the general impression that orange juice is in any case more beneficial. Grape juice retains its aroma but does not have the characteristic blue colour which the consumer expects, and this can only be obtained at the expense of aroma.

Vacuum-hydrated milk, which only occupies one-third of the volume of whole milk whilst retaining its nutritious qualities is now regarded as a commercial failure. Freight charges were considerably reduced

but unexpected resistance was met from trade unions because local deliverymen are paid on a volume basis. Farm combines opposed the products because of the monopoly they had previously held in 'regional' whole milk. Bad city water for diluting the concentrate and the necessity for consuming the liquid within about ten days' time completed the downfall of the product for the time being. Nevertheless, it is predicted that these problems will be overcome and that very large quantities of vacuum equipment will then be required. The authors even state that in the near future all liquid foods will be prepared by vacuum methods and sold as concentrate or powder.

Freeze drying of meat, vegetables and fresh fruit has been quite successful. The products retain their original form and only require the addition of a little iced water for rejuvenation. Transport is considerably simplified as paper cartons can be used instead of wooden crates and refrigeration is unnecessary. This has enabled sections of the community to enjoy the flavour of fresh vegetables where they have not previously been available.

The report is written in a vigorous and entertaining style and should be read by anyone associated with a process industry where the possibilities of a vacuum method may have been overlooked. It is also to be recommended to vacuum technicians in general because of the guidance it gives on the potential value of applications where American industry has already shown the advantages of vacuum methods.

J. BENNETT.

. . . *of vacuum interest*

Lectures

The Acton Technical College, High Street, Acton, London, W.3, announces a course of 12 lectures on BASIC HIGH VACUUM TECHNOLOGY, on Fridays at 6.30 p.m., beginning April 9th, 1954. The lectures will be given by D. J. Pacey, B.Sc., A.Inst.P. Practical work is included. Fee for the course £1 10s.

●

FURTHER LECTURES are announced as follows :

September 21st, 1953 *at 2 p.m.*
Discussion : Freeze Drying Cultures
Chairman : *Lord Stamp*
Held at : The London School of Hygiene and Tropical Medicine, W.C.1.
Arranged by : The British Commonwealth Collections of Micro-organisms

November 17th, 1953, *at 5.30 p.m.*
Freeze Drying
Lecturers : *Dr. R. Kekwick and Dr. R. Strickland-Constable*
Held at : The Institution of Mechanical Engineers, London, S.W.1.
Arranged by : The Physical Society, Low Temperature Group.

November 26th, 1953 *at 7 p.m.*
High Vacua
Lecturer : *A. S. D. Barrett*
Held at : Alliance Hall, London, S.W.1
Arranged by : The British Compressed Air Society

Professional

December, 1953.

United States. AT A MEETING in New York in June last year, a COMMITTEE ON VACUUM TECHNIQUES INC. (CVT) was formed, sponsored by industry, universities and vacuum equipment manufacturers. The new organisation will develop on the lines of a professional technical society. Its immediate task is the standardisation of nomenclature, testing techniques and equipment performance ratings. Messrs. J. B. Merrill, H. Bliven, E. M. Brown, R. A. Koehler, J. H. Durant, B. B. Dayton and F. McNally were appointed chairmen of the Organisation, Finance, Arrangements, Programme, Publicity and Publications, Standards, and Education Committees respectively. The first event the Committee plans is a High Vacuum Symposium at Asbury Park, N.J., in June 1954. Applications for membership from individuals and companies concerned with high vacuum work are invited and should be addressed to Box 1282, Boston 9, Massachusetts.

●

WE CORDIALLY CONGRATULATE Messrs. A. H. Beck and A. D. Brisbane, on the award of a premium by the Radio Industry Council, London, offered to non-professional writers for commendable presentation of technical information. The prize-winning article was published in the April 1952 issue of 'Vacuum' under the title, 'A Cylindrical Magnetron Ionisation Gauge'.

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CLASSIFIED ABSTRACTS

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Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

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All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

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10 — VACUUM ENGINEERING (GENERAL) — 10

Vacuum Technique

174/I

United Kingdom. Recent progress in plant design and processing methods is reviewed. The article is largely based on the lectures presented at the Symposium of Vacuum Physics organised by the Institute of Physics and held at Birmingham in 1950. A continuous vacuum crystallisation process is discussed which is a development of the vacuum evaporation process originally developed for the sugar industry. After the introduction of the warm mother liquor into the evacuated vessel the adiabatic evaporation of the solvent cools the solution to a temperature corresponding to that of the saturated vapour pressure of the solvent and the crystals separate out. Steam ejectors are used, backed by rotary pumps to produce a vacuum of the order of 50–100mm.Hg. The exhaust steam is used for heating the liquor in the evaporator. The work of Hammer, Messing and Schenck is mentioned. Comparative figures for the cost of evaporation cooling and ammonia compression cooling are quoted.

Article by S. L. Martin *Chem. Process Engng.* 33, July 1952 370-373

Sommaire : Etudes sur les développements récents en technique du vide, basées sur les conférences présentées au congrès de Birmingham organisé par l'Institute of Physics.

Much Ado about Nothing

175/I

United States. Four centuries B.C. Aristotle explaining the operation of a suction lift pump said : ' When the piston of the pump is raised water rushes in. Otherwise a vacuum would be created and *Nature abhors a vacuum* '. A cubic inch of air (at sea level) contains 400,000.000.000.000.000.000 molecules. Modern diffusion pumps can reduce this number to the equivalent of one marble left in a container of the size of New York's Metropolitan Opera House. During the war Oak Ridge required diffusion pumps of 30 inch. diameter, 8 feet height and a speed of 20,000 cubic feet of air per minute for the atom bomb project. These wartime developments facilitated the post-war progress of vacuum technique to the present stage. Today, vacuum methods are established in industry for the deposition of metal on plastics, glass, fabrics and sheet materials. Synthetic fibres can now be dyed in a high vacuum. Leather is impregnated in a vacuum. Vacuum processes yield steels and alloys of hitherto unknown purity, density and ductility. The vacuum engineer himself is helped by these new materials as they facilitate the design of pumps and vacuum vessels for steadily lowered ultimate pressures. The Brookhaven cosmotron is a typical example. Its magnet has a diameter of 60 feet and is assembled from 288 flat steel blocks each 8 feet high, 8 feet wide and weighing nearly six tons. The vacuum chamber situated in the gap of the magnet is built of one inch. thick stainless steel side walls which are bolted together by stainless steel bars each about two inches wide. Twelve 20-inch diffusion pumps connected to 16-inch valves produce the vacuum required.

Article by Anon. *Steelways* 8, Sept. 1952 5-7 see also *Engineer*, 195, 12.9.52 347

Sommaire : Revue des développements de la technique du vide dûs aux nécessités de la guerre.

Method for the Production of a Vacuum

176/I

See Abstract No. : 126/II

Working Principle of the Vacuum Pumps

177/I

Australia. A theory is proffered asserting that a common pumping principle is the basis of every vacuum pump, by producing a low pressure in a limited space and removing the gas molecules over a gradient of increasing gas density.

(*B.S.I.R.A. Bulletin*)

Treatise by N. A. Florescu *Invest. Phys.* No. 1, 1952 8 pp.

Sommaire : On donne une théorie concernant les principes de fonctionnement communs à tous les types de pompes à vide.

A Summary of the Present Status of Flow and Heat Transfer in Rarefied Gases

178/I

United States. The paper summarises the present experimental and theoretical knowledge of the effect of low gas densities on flow and heat transfer phenomena. This subject gains steadily in importance since projectiles are now used for the exploration of the upper atmosphere reaching the height of 100 miles, where the molecular mean free path is of the order of 10 feet. At sea level the molecular mean free path is of the order of 10^{-7} feet and in this range the media which transfer the heat are considered continua. This concept is inadequate at low pressures. Tsien proposed to distinguish between three pressure-defined types of flow which show an analogous difference of heat transfer characteristics as follows : (1) the continuum region, where the mean free molecular path is small compared with a characteristic dimension, (2) the free molecular flow region, where the mean free path is large compared with a characteristic dimension and (3) the transition region where the mean free path is small but not negligibly small compared with a characteristic dimension. Region 1 is well explored. The continuity momentum and energy equations of laminar flow are known and offer a means of analysis. Region 2 is partly explored and can be analysed with the help of the kinetic theory of gases. Region 3 is little explored and no complete analysis is available. These considerations apply essentially to flow and heat transfer by convection. As the pressure is reduced heat transfer by radiation becomes

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increasingly dominant. Since no direct solutions are available in region 3 it has been proposed to use the continuum formulations of the flow and energy equations for an analysis and relegate the rarefaction effects to the boundary conditions. Two relevant effects have been noted in the slip flow region : (1) the flow near a boundary is not at zero velocity at the wall but slips over the surface with a finite velocity and (2) a temperature discontinuity or ' jump ' exists at the wall during heat transfer from a surface to a rarefied gas. These effects can be represented by mathematical expressions linking the slip flow boundary conditions to the energy characteristics of the surface. With the help of the Rayleigh flow equation a solution can then be obtained for the skin friction on a flat plate. This solution has the correct form but differs in numerical value from both extremes, continuum and free molecular flow. A similar analysis is presented for the heat transfer from a flat plate. Analysis of the heat transfer from cylinders and spheres are also given. The authors proceed to discuss heat transfer at free molecular flow conditions with a similar approach and report the results of experimental work. The investigations were carried out in the low-density supersonic wind tunnel at the University of California, Berkeley.

Report by R. M. Drake, Jr., S. S. A. Schaaf *O. N. R. Monthly Report* July 1951 8-15

Sommaire : Résumé de l'état actuel des connaissances théoriques sur l'écoulement et la transmission de la chaleur dans des gaz raréfiés.

Drag on a Rotating Cylinder at Low Pressures 179/I

United States. The torque transmitted by a rarefied, laminar air flow between two concentric cylinders for a case where the inner cylinder was rotating while the outer one was held stationary was investigated. A sensitive torque balance was designed and with it the torque on the stator (drag cylinder) was measured at various speeds and pressures. In this work the ratio of Mach number to Reynolds number (M/Re) plays the same role as the Knudsen ratio of mean free path to dimension of apparatus (λ/h). The experiments covered the ranges in Mach number from 0.14 to 0.55, pressure 220 microns of mercury to 1 micron and hence λ/h from 0.0176 to 3.87 corresponding to M/Re from 0.0119 to 2.61. Within these ranges the experimental data showed that the rotor aerodynamic drag was proportional to the speed of rotation and the reduction of drag at low pressures was independent of Mach number. The instability of laminar flow was also tested. The critical Reynolds number was found to be 135. No effect of compressibility or slip on the critical Reynolds number was observed. A theoretical analysis was made to obtain the differential equations, boundary conditions, and particularly the drag coefficient of a rarefied Couette flow (steady, two dimensional viscous flow between two parallel plates with relative motion). The thirteen moment method of H. Grad was used and the results compared with those of R. Schamberg. It was found that (1) the slip boundary theory with specular reflection coefficient f=0.9 agreed with the experimental results at all test speeds and pressures. (2) The drag coefficient expressions derived by the thirteen moment method and Schamberg's analysis differ only in third order terms. (3) The difference between the drag coefficient values obtained by slip boundary theory, Schamberg's theory and thirteen moment analysis is less than 1% and cannot be determined by experiments in the slip flow regime for M<1. (4) The theoretical critical Reynolds number is about 7% lower than the experimentally determined value.

Report by Shih-Fei Chiang *Univ. Calif. Tech. Rep. No.* HE-150-100 19.5.1952

Sommaire : La torque effectuée par un courant d'air laminaire en atmosphère raréfiée, entre deux cylindres concentriques est étudiée pour le cas dans lequel le tube intérieur tourne et l'extérieur est immobile.

Nozzle Characteristics in High Vacuum Flow—Rarefied Gas Dynamics 180/I

United States. Knowledge of the performance characteristics of flow measuring devices for rarefied gases, *i.e.* for flow under low Reynolds number conditions becomes increasingly important. Low Reynolds numbers exist in a flow system where the density or pressure of the gas is reduced to a small value. Assuming that the ambient temperature and velocity in a system is kept constant a Reynolds number of 100,000 at atmospheric pressure is reduced to 100 at 0.001 atmosphere. Differential metering devices operating with fluids at such low Reynolds numbers show a much reduced flow coefficient. This is due to the fact that the thickness of the boundary layer in the nozzle increases as the Reynolds number decreases, resulting in a change of the flow velocity distribution at the upstream position and at the throat. With the help of the Knudsen number, λ/D, the ratio of the molecular mean free path to the nozzle throat diameter, the pressure conditions at normal temperatures determining the nozzle throat performance can be divided into the following three regimes : continuum flow (Kn smaller than 0.01), transition or slip flow (Kn equal 0.01-15) and free molecular flow (Kn greater than 15). In tests with air at atmospheric pressure keeping temperature and pressure at the upstream position constant and varying the downstream pressure at ratios of down- to up-stream pressure below the critical value of 0.528, it was found that the weight rate of discharge was constant and independent of the downstream pressure. Corresponding initial experiments at very low pressures demonstrated that the rate of flow was not constant. The discharge coefficient (the ratio of actual to theoretical weight rates of the flow to the nozzle) decreased as the boundary layer thickness increased. A series of experiments was then carried out to explore the conditions in the slip flow region. Details of the nozzles tested and the equipment used are given. A low-pressure wind tunnel was employed. The nozzle under test was placed between the upstream chamber and the down stream (test) chamber. The air passed through the nozzle and test chamber into a ballast tank which was connected to the vacuum pump. All tests were carried out at a pressure of approximately 0.001 mm. Hg. A McLeod gauge was used for pressure readings. The volume of air flowing through the apparatus was measured with the help of a gasometer. The results of the experiments are shown in graphs Generally the values showed the trend expected but the plottings of the discharge coefficients against the throat

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Reynolds number exhibited certain characteristic deviations from a continuous line as the Reynolds number increased and the pressure ratio decreased. These break-away points appeared to be typical for the slip flow region. To investigate these results, further tests were made with one nozzle comparing the pressures at the throat with those of the downstream chamber. The pressures at the throat proved to be higher than the corresponding values in the downstream chamber. Thus it appeared that the pressure in the thick boundary layer was not of the magnitude expected and may have differed from those at corresponding positions in the core of the fluid. Consequently the expansion ratios and velocity distributions would differ and this would show as a change in flow coefficient for otherwise similar flow conditions.

Sommaire : Les performances de trois ajutages de construction définie ont été étudiées dans le domaine de l'écoulement semi-moléculaire.

Article by R. G. Folsom *Trans. A.S.M.E.* **74**, 1952 915-918

A High Vacuum Flow Analogy

181/I

United States. The Knudsen equation for the mass flow of gas through a tube under high vacuum conditions can be written as follows :—

$$N = \frac{8\pi R^3}{3L}(\nu_1 - \nu_2)$$

where N is the molecules per second flowing down a cylindrical tube of the length L and the radius R as a function of the difference in the number ν of molecules striking a square centimetre per second at the two ends of the tube. Supposing that the molecules travel at fixed speeds, do not interfere with each other and are reflected from the tube wall at random angles analogous conditions should apply, if electro-magnetic energy is conveyed through the tube as long as its wall is highly reflecting and each photon carries the same energy. The electro-magnetic energy P conducted through the tube can be expressed as follows :—

$$P = \frac{8\pi R^3}{3L}(I_1 - I_2)$$

where I_1 and I_2 are the intensities at points which are a distance L apart. Thus electro-magnetic conductance measurements could simulate conditions of high vacuum flow and allow the assessment of pumping speeds before the vacuum system is finally built. In order to determine the conductance $C = 8\pi R^3/3L$ a uniformly illuminated ground glass screen could be employed as a light source which allows light to enter into and pass along a tube lined with crinkled aluminium foil and identical in dimensions to the vacuum pipe. Through small holes in the wall the intensities could be measured photoelectrically. The loss of light intensity along the tube facilitates an evaluation of the conductance for gas flow.

Sommaire : Formule applicable à la propagation d'énergie électro-magnétique à travers un tube analogue à l'équation de Knudsen pour l'écoulement de masse d'un gaz à travers un tube sous condition de haut vide. Celle-ci peut être utilisée pour l'exploration d'une installation à vide projetée par le mesurage de l'intensité lumineuse transmise à travers un modèle de la tuyauterie en projet.

Note by G. W. Monk *J. Appl. Phys.* **23**, July 1952 799-800

Clean-Up of Helium Gas in an Arc Discharge

182/I

United States. Gas clean-up, or the gradual disappearance of the filler gas in an electrical discharge, has been investigated previously by Pietsch and Alterthum, Lompe and Seeliger. In the present experiments, based on the work of the authors quoted, it was attempted to obtain a controlled clean-up, bombarding a tantalum surface with helium ions. Special precautions were taken to make sure that highly purified gas only was employed. An oxide cathode of the ordinary commercial type was used with a nickel mesh base and an area of about 30 sq. cm. The anode was of nickel and of the hollow cylindrical type. Two different probes were used, one consisting of a tantalum wire 0.01 inch. diameter mounted so that current could be passed through it for degassing purposes. Only a fraction of its length was exposed to the discharge. The other consisted of an open-ended cylinder 1.55 inch. wide and 1.55 inch. long, with a wall thickness of 0.005 inch. The latter could be moved and in one position could be heated to high temperatures without affecting the glass envelope. The number of disappearing helium atoms was determined by measuring the pressure drop in the system. Allowance was made for other sources of clean-up present, *e.g.* the gas leakage through the tube wall which is independent of the presence of a discharge and for which data exists. The most important type of self clean-up is that into the cathode. Its rate depends on the arc drop. With a good cathode the arc drop could be maintained at a value for which the self clean-up rate was approximately 10^{-2} mm. Hg per minute. Poisoning of the cathode caused the arc drop to rise resulting in an increased self clean-up rate. The tube was operated with a pulsed negative potential on the probe, *i.e.* with an ion current to the probe. Tests proved that the probe clean-up occurred invariably at a rate 10 × as high as that of the self clean-up. The results of the experiments are shown in graphs. Two diagrams giving the ratio of ions cleaned up to ions striking the probe against probe potential demonstrate that the number of ions cleaned up is very largely a function of probe potential. In the case of the wire probe 40-85% were trapped and in the case of the cylindrical probe 4-40%. In order to examine clean-up phenomena associated with sputtering a reversal of the clean up process was attempted. After heating the probe wire to 2,000°C a volume of gas equivalent to 5-22% of the net probe clean-up was recovered. Heating the cathode to 825°C, with the tube dark, reproduced 11-54% of the amount absorbed during self clean-up.

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Heat treatment of the tube envelope yielded 8-31% of the gas which had disappeared due to all sources of clean-up. In the case of the cylindrical probe recovery amounted to 70% and the nearby region of the glass envelope yielded 35% out of 70% which was recovered from the overall area of the envelope. No explanation could be found for the fact that only a small amount of the cleaned up gas could be recovered from the wire probe. The ions may have been reflected as neutral atoms and trapped on striking a nearby surface.

Sommaire : Etude méthodique des phenomènes d'abbaissement de la pression du gaz dans des tubes à décharge.

Article by M. J. Reddan & G. F. Rouse *Trans. A. I. E. E.* **70**, Pt. 2, 1951 1924-9

The Principles of Leak Detection

183/I

United Kingdom. The report discusses dynamic methods of leak detection where the surface of a continuously evacuated system is probed with a jet of some gas or vapour which is not initially present to any appreciable extent in the system. Leak detection with the detector on the high vacuum side and on the backing side of the main diffusion pump is discussed and equations are derived for the partial pressures of air and probe gas in the system at any time. Optimum leak detection conditions are determined and data are given enabling the minimum detectable leak to be calculated when certain detectors and probe gases are used in any system. (*Author*)

Sommaire : Rapport sur les méthodes dynamiques de détection de fuites.

Report by A. H. Turnbull *A.E.R.E.* Rep.No. G/R478 March 1950

Studies in High Vacuum Evaporation. The Falling Stream Tensimeter

184/I

ee Abstract No. : 206/III

Studies in High Vacuum Evaporation. Projective and Equilibrium Vapour-Liquid Relationships for Two Binary Systems Over an Extended Range

185/I

See Abstract No. : 207/III

Studies in High Vacuum Evaporation. Surface Behaviour in the Pot Still

186/I

See Abstract No. : 208/III

Studies in High Vacuum Evaporation. Comparison of High Vacuum Stills and Tensimeters

187/I

See Abstract No. : 209/III

The Molecular Distillation of Fats and Oils

188/I

See Abstract No. : 205/III

Study of Vacuum Fumigation Techniques

189/I

See Abstract No. : 197/III

The Method of Vacuum Evaporation

190/I

See Abstract No. : 173/III

Vacuum Courses at the Royal Technical College, Glasgow

191/I

United Kingdom. A Report is given on the purpose, and curriculum of the Vacuum Course inaugurated shortly after the war at the above College. There is a day course designed to be an integral part of the 4-year course for the Associateship of the Royal Technical College and an evening course originally established to suit students who wished to take the Higher National Certificate in Applied Physics, but now being modified to post-graduate standard suitable for endorsement on the Higher National Certificate. Details are given of the equipment available and the practical laboratory work. The evening course takes 10 weeks.

Sommaire : Détails de l'historique et de l'équipement à disposition d'un cours de vide ayant débuté après la guerre au Royal Technical College de Glasgow.

Report by C. Weaver *Vacuum*, 2, Oct. 1952 377-381

Lectures on 'Industrial Applications of High Vacuum Technology'

192/I

United Kingdom. A brief documentation of last years special lecture course, organised by Northampton Polytechnic, London, under the above title. Abstracts of each of the lectures are given emphasizing, and in some cases amplifying, the more important aspects of the information presented in the original paper.

Sommaire : Objets significatifs de conférences publiées l'année dernière sous les auspices du Northampton Polytechnic à Londres sur l'application industrielle de la technique du haut vide.

Report by Anon. *Vacuum*, 2, Oct. 1952 381-387

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13 — ELECTRICAL SCIENCES — 13

Failure of Vacuum Insulation at Low Field Strengths — 193/I

See Abstract No. : 83/IV

The Effect of Surface Treatment on Electric Breakdown Between Copper Electrodes at 50 Cycles at Very Low Pressures — 194/I

United Kingdom. This memorandum describes the preparation of pure copper electrodes prior to electric breakdown measurements at 50 cycles/second at very low pressures. An account of the electrical measurements and the results obtained is also included.

(*Author*)

Sommaire : Recherche sur l'effet du traitement de surface sur l'effondrement du vide aux très basses pressions.

Report by R. J. B. Hadden *A.E.R.E.* Rep. No. G/M92 Aug. 1951

The Threshold Gas Pressure Required to Sustain a Stable Arc in a Magnetic Field — 195/I

United Kingdom. The presence of a longitudinal magnetic field increases the efficiency of ionisation in a discharge at low pressures. Therefore the minimum pressure required to maintain an arc is reduced. The author investigates the minimum threshold pressure required for pulsed high current arcs between tungsten rod electrodes in air and hydrogen. The discharge tube used in the experiments was 3.2 cm wide and the tungsten rod electrodes were 5.1 cm apart. In synchronism with the first half cycle of the magnetic field pulse a cold cathode discharge of 96 amps was initiated between the electrodes. The pulse lasted 2 millisecs. The breakdown of the gap was achieved by applying a steady 25,000v initiating potential through a high resistance to the anode. In hydrogen the lowest pressure at which the arc could be maintained was 3.2×10^{-3} mm.Hg. In the case of air the minimum pressure was 2.05×10^{-4} mm.Hg. The minimum magnetic field strength was 1,475 gauss and 2,000 respectively. The author compares his results with those of Burhop who used lower current arcs in helium. According to the formula developed by Burhop and quoted in this article the threshold pressure for hydrogen would be 3.0×10^{-3} mm. Hg and for air 1.5×10^{-3} mm. Hg. Discussing ionisation phenomena the author states that re-combination in the arc column was improbable as the electrodes were not sufficiently far apart. But, if extensive re-combination in the area near the cathode took place an appreciable pressure increase may have occurred locally, resulting in a pressure gradient along the axis of the tube and part of the arc column may have been in a lower gas pressure than the threshold value. Also it was found in the hydrogen experiments that clean-up reduced the pressure to less than 2.7×10^{-3} mm. Hg during one pulse. The fact that the arc was maintained was probably due to the presence of vapours near the electrode originated by extensive sputtering.

Sommaire : Recherches sur la pression de seuil permettant de maintenir un arc stable dans de l'air ou de l'hydrogène, par cela amplifiant les résultats obtenus par Burhop dans ses recherches sous atmosphére d'hélium.

Article by N. L. Allen *Proc. Phys. Soc.* **65.B**, Sept. 1952 697-701

Method and Apparatus for Fusing Dielectric Material in a Partial Vacuum by High Frequency Electric Fields — 196/I

United States. The invention described represents a development of the hot bar sealing method. The method is applied in packaging food which tends to deteriorate in the presence of air and facilitates the sealing of bags made from thermo-plastic film such as cellophane, in a vacuum. The apparatus consists of a vacuum chamber containing the sealing bar which consists of bare metal and can be moved up and down. The bar constitutes one of two electrodes and is connected to the body of the chamber. The other electrode is fixed. But all its faces except the one which makes electrical contact with the former are insulated. The electrodes are connected to an a.c. power supply of 500 V 60-120 megacycles. In operation the bag containing the food is placed in the chamber with its open end between the electrodes, the chamber is evacuated to a pressure of 25 inch. mercury and the sealing bar is brought down. Then, the power is switched on for the required period. Because of the time element in food packaging procedures a lower pressure cannot be provided. But in order to ensure satisfactory operation of the apparatus, it is desirable to cover all electrode faces with insulating material and hereby to provide a thermal buffer as is the practice in heat sealing at atmospheric pressure. This is possible in the vacuum process, if the voltage of the supply is stepped up and if all non-conductive faces of the electrodes carry insulation of a thickness equivalent to that of the insulation on the conductive-faces plus the depth of the effective field created by the electrical charge.

Sommaire : Méthode de scellement de sachets contenant des denrées alimentaires par méthode électronique sous vide.

E. Zimmerman *Brit. Pat.* 672,258

A Demountable Tetrode Ionisation Gauge — 197/I

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Ionisation Gauge — 198/I

See Abstract No. : 134/II

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14 — MECHANICAL SCIENCES — 14

Design Features of Modern Vacuum Drying Plant

199/I

Germany. The walls of an evacuated container have to withstand a pressure of 10 tons per square metre. If the container is rectangular the walls must be especially thick or well reinforced. Therefore cylindrical shapes are used wherever possible. But in the case of the normal shelf dryer a rectangular shape is preferable for functional reasons. The volume of cylindrical drying cabinets is up to 50% greater than that of a rectangular drying cabinet of the same shelf capacity. This results in longer pumping times, or, alternatively, if standard pumping times have to be adhered to (5 minutes for heat sensitive materials and 10 minutes for other materials) in bigger and more expensive pumping units. Another point to consider is corrosion. The moisture is extracted from the processed material in the form of steam. If the steam contains active components it will induce corrosion wherever it has a chance to condense. In cylindrical dryers the heat transfer from the shelves to the walls is comparatively ineffective and it is a common occurrence that condensate collects at the bottom. In the past, rectangular vacuum dryers were made from cast iron. Now welding techniques have improved sufficiently to make the use of steel possible. An important factor governing the design of shelf dryers is the quantity of material to be dried which it is intended to place on the individual shelves. If the material has a high moisture content and good thermal conductivity, drying time will increase in direct proportion to the thickness of the layer, but if the material is fairly dry and has a low thermal conductivity drying time will increase at the rate of the square of the thickness. In practice the layers of the processed material are approximately 10 mm. thick. The spacing of the shelves is largely determined by the nature of the material to be processed. If it tends to foam the spacing should be wide. In all other cases the spacing should be as close as can be managed considering the manipulation of the shelves. The closer the spacing, the better use is made of the heat radiated from the upper shelf. The table reproduced below demonstrates the effect of shelf size and spacing on the amount of heat transmitted by radiation for the case of heating by steam and a heat transfer coefficient of the processed material k = 5.

Size of Shelves	*Heat Transmitted by Radiation in % of Total at a Spacing of the Shelves as Shown*		
m × *m*	150 *mm*	100 *mm*	50 *mm*
1 × 1	48	50.4	51.8
1,5 × 1	50	52.5	54

Sommaire : Description de problemes techniques liés à la construction et la fabrication d'installations de sèchage sous vide, en particulier des dessicateurs à plateaux.

Article by H. Haas *Z. V. D. I.* 94, May 1952 357-359

15 — PHYSICS — 15

High Altitude Research

200/I

United Kingdom. A detailed description is given of the operation of long-range rockets in the United States for the exploration of the upper atmosphere. Three types of rockets are mentioned, the modified German V2, the Aerobee and the Viking. Details are given of the instrumentation used and the operation and actual arrangement of the instruments in the nose of the rocket. Some of the measurements were telemetered to the ground during flying. Details of the transmission system are given. In order to preserve records part of the equipment was jettisoned by parachute shortly before landing. The pressure of the atmosphere was measured in three ranges (*a*) from atmospheric to a few mm. Hg by means of a simple bellows gauge, (*b*) from a few mm. Hg to 10^{-1}mm. Hg by a Pirani gauge and (*c*) between 10^{-2} and 10^{-8} mm. Hg by ionisation gauges. The former two gauges were mounted just ahead of the tail fin. The latter were fitted with a connection between the envelope and the outside of the rocket. During flight at low altitude the ionisation gauges were sealed. At the correct height the seal was broken by a timing device. Four ionisation gauges were used in each rocket. The pressure gauge described in Vol. I (Abstr. 54/II) has also been used and satisfactorily measured the pressure from 660 mm. Hg at ground level to 570 μ Hg at 53 km. altitude. The temperature of the upper atmosphere was calculated from the pressure data. The Mach numbers were obtained from the observed speeds of the rocket facilitating calculations of the local speed of sound. Attempts have also been made to ascertain the composition of the upper atmosphere. Evacuated bottles were used which opened and closed automatically, controlled by a timing device. Analysis of the samples obtained indicated gravitational separation with the lighter gases rising to the topmost regions. Cloud chambers were flown in the rockets to measure cosmic radiation. The peculiar conditions arising from flying cloud chambers at high altitudes are discussed in detail. Visual spectra were obtained with a solar spectrograph and the relative spectral intensities determined. The modified V2's reached a height of nearly 120 miles, the Viking 135 miles and a more recent design of the V2 250 miles.

Sommaire : Description détaillée de la méthode employée pour l'étude des conditions dans l'atmosphère supérieure a l'aide de fusées, avec référence particulière à l'instrumentation d'enregistrement de pressions.

Article by E. Burgess *Engineer* 195, 12.9.52 338-340 & 195, 19.9.52 370-373

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An Analysis of Heat Transfer through Thermal Insulating Material

201/I

Canada. The efficiency of heat insulating materials has been investigated. The experiments were carried out on an 8 inch. guarded hot-plate built in accordance with the specifications of the Bureau of Standards and enclosed in a cylindrical tank, the air pressure of which could be varied from 80 inch. Hg to 0.1 inch. Hg. Radiation was investigated first. The results of tests are given in Table I reproduced below in abbreviated form. It is shown that the air cells tested which had a size of 8 × 8 × 1 inch transmit 74% of the total heat by radiation (after allowance has been made for the emissivity of the metallic foil).

Table I

Specimen	*Mean Temperature °F.*	*Temperature Difference °F.*	*Conductivity k*
Air Cell :			
Paper Surfaces	82.0	25.7	1.06
Aluminium Foil Surfaces	90.7	36.0	0.278
Aluminium Foil Surfaces	76.2	40.8	0.276

Investigating convection the author employed the method of varying the air density in the pores or cells of the insulating material. As a preliminary measure cells of the same size as before, faced on both sides with aluminium foil were tested, filled and unfilled, at different air pressures with the results as shown in Table II reproduced below.

Table II

Specimen	*Air Pressure inch. Hg*	*Mean Temperature °F.*	*Temperature Difference °F.*	*Conductivity k*
Air Cell (vac) :				
Both sides faced with aluminium foil	29.94	76.2	40.8	0.276
	24.27	83.1	48.6	0.249
	9.06	83.1	44.2	0.225
	4.44	85.4	49.9	0.212
	2.69	92.3	60.1	0.206
	1.72	96.4	57.8	0.192
	1.18	98.1	63.5	0.191
	0.294	92.9	55.2	0.202
	0.220	92.1	59.8	0.200
	0.153	94.0	67.4	0.192
" Styrofoam " :				
Density 1.34 lb./cu. ft.	29.9	81.4	33.1	0.307
	1.0	80.7	34.3	0.301

Among the various powders of small grain size investigated was Santocel, a Silica Aerogel powder with black filler. Results are shown in Table III (see below).

Table III

Specimen	*Air Pressure inch. Hg*	*Mean Temperature °F.*	*Temperature Difference °F.*	*Conductivity k*
" Santocel " (SAN) (Silica Aerogel) (with Black Filler) : Density 3.88 lb./cu. ft. Particle Size 20-40 Mesh (U.S. Standard)	29.72	107.0	61.4	0.185
	21.5	103.3	65.8	0.179
	12.7	107.3	67.9	0.170
	9.28	108.0	73.2	0.162
	2.77	106.9	80 2	0.155
	1.84	112.0	76.4	0.149
	0.23	125.8	116.7	0.104

The conductivity of 0.185 obtained at atmospheric pressure is very close to that of still air as given by Keenan and Kaye. Similar tests were made on glass wool of various densities and on tightly packed wax-coated drinking straws. Previous investigations indicated that, if the size of the cells was large as compared with the mean free path of the gas, the reciprocal of absolute pressure plotted against the reciprocal of conductivity

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gave a straight line. This was confirmed by experiments in air up to a pressure of about 8 inch. mercury. Beyond that pressure there was an increasing deviation from the straight line relationship owing to the increasing influence of convection. Kannuluik and Martin working on powders confirmed these conditions and developed the formula $p/k = ap + b$ where p is the absolute pressure of the gas, k is the thermal conductivity and a and b constants. Plotting the curve of p/k against p and drawing a straight line tangentially to the lower end of the curve gives the heat transmitted by convection as the distance between test curve and straight line. The curves drawn fror Santocel were practically straight at higher pressures indicating that there is no appreciable transmission by convection. This is probably true for most fine powders. The results reported here apply only to the size of air cell employed in these experiments. The effects of convection would probably be greater if larger air cells were used.

Sommaire : Rapport des essais de laboratoire utilisant la technique de vide pour la determination de la valeur des différents types de matériaux isolants à la chaleur.

Article by E. A. Allcut *Engng. J.*, **35**, Aug. 1952, 836-841

On New Methods of Insulating Very Low Temperature Apparatus

202/I

Russia. Partially evacuated " mipora ", an expanded urea formaldehyde plastic (density 18-22 kg/m³, porosity 97-98%) combines the cheapness and durability of solid insulation with the excellent properties of high vacuum as thermal insulation for low-temperature apparatus and containers for liquid, O, H, N, CH, etc. Plotted test results show the rates of evaporation of liquid air and liquid H from a cylinder insulated by high vacuum (10^{-5}mm. Hg), partial vacuum " mipora " at atmospheric pressure, evacuated " mipora " (to 10^{-2}mm. Hg for liquid air, and 0.2 mm. Hg for liquid H) and " mipora " at liquid air temperatures from 0.022 to 0.0025 kcal/m hr°C.

(*Science Abstracts*)

Sommaire : Nouvelles méthodes d'isolement d'appareil a très basse temperature.

Article by A. B. Fradkov. *Dokl. Akad. Nauk SSSR* **81**, No. 4, 1951 549-51

An Optical Cell for Use with Liquid Helium

203/I

United States. The authors describe a cell specially developed for the study of the optical properties of alkali halides and the absorption spectra of materials which could be supported between or deposited on alkali halide crystals. The cell is 6 inch. wide at the top but narrower at the bottom so that the sample is only 1 inch from the outside window. It consists of three sections, an outer vacuum jacket including the windows, a liquid nitrogen container and radiation shield and a liquid helium container and sample holder. A common vacuum is maintained between all sections. The liquid helium container is the innermost section, holding 1 litre of helium and is suspended from a stainless steel tube $\frac{5}{8}$ inch diameter and 17 inch. long which is soldered to a collar fitted to the top end plate. Concentric to the latter is a stainless steel tube of 1 inch diameter, the lower end of which terminates in a sylphon bellows. This tube can be raised and lowered by means of a crank rack and pinion assembly and by this means the helium container can be raised and lowered by approximately 2 inch. The nitrogen container is suspended from the top plate of the assembly. At the lower end is a copper heat shield which is kept at 77°K by conduction cooling. Except for the radiation through the windows the liquid helium vessel ' sees ' nothing warmer than that. The outer housing is made from stainless steel. After evacuating the cell to 10^{-4} mm. Hg liquid nitrogen is filled into the nitrogen container and, for the purposes of pre-cooling, into the helium container. At this stage the pressure should have dropped to 10^{-5} mm. Hg. The cell is then removed from the vacuum line, the liquid nitrogen in the helium container boiled away and helium filled in directly from a Collins liquid helium cryostat. The cell is fitted with rock salt windows and one beryllium window. The crystal holder can carry two samples. A different design is described for use with samples in vapour form. Liquid samples, too, can be used. In ordinary circumstances one charge of helium will last for about 20 hours. One filling of nitrogen, approximately 1.5 litres, lasts 12 hours. The outside of the cell remains at room temperature during operation, and if the cell is sufficiently vacuum tight, the liquid helium container acts as the vacuum pump thus making the cell completely portable.

Sommaire : Cuve optique pour mesure aux basses températures dans le domaine de la spectroscopie infrarouge.

Article by W. H. Duerig & I. L. Mador *Rev. Sci. Instrum.* **23**, Aug. 1952, 421-424

Filters for the Infra-Red Region

204/I

See Abstract No. : 192/III

Theoretical Study of the Interference Filter

205/I

France. The solution is given of the problem of the propagation of a plane monochromatic light wave through an interference filter consisting in a periodic system of any number m of double layers with indices n_1 and n_2 and thicknesses d_1 and d_2 without taking account of absorption. The transmitted intensity (when $n_1 d_1 = n_2 d_2$ for normal incidence) is expressed as a function of wavelength. By superposition of two of these filters, one can obtain a practically monochromatic filter of high transmittance.

(*Science Abstracts*)

Sommaire : Etude théorique du mécanisme de fonctionnement de filtres interférentiels.

Article by D. C. Frau *Rev. Opt. (Theor. Instrum.)* **31**, April 1952 161-169

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A Tracer Method for the Thickness Measurement of Thin Bi Films

206/I

United States. For the study of very thin bismuth films deposited by vacuum evaporation very accurate values of the average thickness of the films were required. The surface of molten bismuth tends to be covered with a layer of the oxide and this affects the quantity of the metal actually evaporated. As none of the known methods of thickness measurement appeared to fulfil the requirements, a method was specially developed as follows : Bismuth containing Bi^{210} was evaporated onto a small piece of glass in a layer of about 300 Å. The deposit was weighed on a sensitive balance and used as a standard. The film to be measured was placed before a Geiger-Mueller counter and the number of counts per minute recorded. Then the standard was placed in front of the counter. The ratio of counts of the unknown to the standard film represented the ratio of the respective weights of bismuth. Determining the absolute weight and the area of the unknown deposit then yields the average film thickness. Details are given of the preparation of the radio-active bismuth used in the experiments. While the material was fresh, chiefly beta particles were counted. Subsequently, for a period of about 12 months, mainly alpha particles were counted. The gamma activity was weak. Special safety precautions were taken. The temperature of the evaporation source was kept constant. The material was made to condense on surfaces which could be cleaned easily and a dry ice and acetone cold trap between diffusion and fore pump retained all vapours before they could reach the atmosphere. No more than 0.1 mg of the material was required for the production of a standard. Two types of deposits were obtained : One more active showing a dull surface and one less active showing a mirror-like surface. Examination in the electron diffraction camera proved that the mirror-like standards consisted of small bismuth particles forming a smooth and uniform surface, whereas the more active dull looking standards showed considerable aggregation of bismuth crystallites. It appeared that the latter surface resulted from poor vacuum conditions in processing. When the necessary adjustments had been made, films of the less active type were always produced. A graph is shown demonstrating that the counting rate *versus* film thickness was linear in the range of O-225 Å. At greater thickness individual plottings were required in order to assess the thickness accurately.

Sommaire : La mesure précise de l'épaisseur de films de bismuth est facilitée par l'usage de bismuth radioactif.

Article by J. J. Antal & A. H. Weber *Rev. Sci. Instrum.* **23**, Aug. 1952 424-426

Measurement of the Thickness of Transparent Films with the Light-Profile Microscope

207/I

United Kingdom. The authors report briefly on the application of Tolansky's light-profile microscope technique for the determination of the optical thickness (t) of relatively thick transparent films. A transparent film bounded by media with refractive indices (n) differing from that of the film has two reflecting surfaces. In a light-profile microscope the two surfaces produce a double image. Separating the two the ratio t/n can be derived. By means of direct interferometry using fringes of equal chromatic order the value n·t can be obtained. Combining the two methods n and t can be determined. If the films are more than a few microns thick the depth of focus prevents simultaneous focussing of both images. The authors overcome this by employing an intermediate focal plane so that both images are defocussed by the same amount. The method has been successfully applied for the determination of local variations in thickness of thin collodion, Canada balsam and liquid films on crystal surfaces.

Sommaire : Brève note sur l'adaptation de la technique du microscope à profilation optique, pour la détermination des épaisseurs optiques de films transparents relativement épais.

Letter by H. Rahbek & M. Omar *Nature* **169**, 14.6.52 1008-1009

The Distribution of Thin Films Condensed on Surfaces by the Vacuum Evaporation Method

208/I

United Kingdom. The problem of determining the laws of distribution of thin films deposits evaporated in vacuum is discussed in detail. From various types of practical sources idealised sources are evolved for which the emission of vapour can be calculated. As a fundamental law, the ' cosine law ' of Knudsen is recognised. The calculation is extended to point sources, long strip sources, wire sources and ring sources. Evaporation on to plane and spherical receiving surfaces is considered. The practical application of these results to the evaporation of zinc sulphide and magnesium fluoride from strip heaters, and aluminium from spiral filaments is considered together with experimental confirmation. The range of validity of the results is discussed.

(*Authors*)

Sommaire : Etude des lois déterminantes la distribution de films deposés par évaporation sous vide.

Article by L. Holland & W. Steckelmacher *Vacuum* **2**, Oct. 1952 346-364

Electron Diffraction in Some Cubical Crystals

209/I

India. The spacings of a number of cubical crystals, obtained by metal evaporation in high vacuum, have been determined in the electron microscope from the diffraction photographs. Except for AgBr, the valves tally with those obtained from X-ray diffraction measurements. The diffraction photographs obtained are very much sharper than similar pictures obtained with metal foils by previous workers.

(*Science Abstracts*)

Sommaire : Les espacements d'un certain nombre de cristaux cubiques après déposition sous vide ont été etudiés sur des diffractogrammes.

Article by S. K. Majumdar *Indian J. Phys.* **25**, Nov. 1951 533-538

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Dissociation of Molecular Hydrogen Ions in the Cyclotron

210/I

United Kingdom. The Birmingham 60-inch. cyclotron is normally used to accelerate molecular hydrogen ions H_2^+, deuterons D^+ or alpha-particles He^{2+}. The same magnetic field is used in each case. In the case of H_2^+ ions the circulating beam current was found to be some 10 to 15 times less than expected. It appeared that the H_2^+ ions dissociated as a result of collisions with residual gas molecules. The energy required for the dissociation of an H_2^+ ion is small and thus collisions would lead to the formation of a proton and a neutral hydrogen atom or two protons, each of half the incident energy. The newly formed protons would move in orbits of half the radius of that of the orbit of the incident H_2^+ ions and eventually strike the ion source at the centre of the cyclotron. This was proved to be the case as the tip of the ion source became white hot when H_2^+ ions were accelerated. If the pressure in the tank was increased more H_2^+ ions were dissociated and the ion current decreased accordingly. Therefore the dependence of ion current on gas pressure could be used to measure the dissociation cross section. Measurements were taken at distances of 18 inch. and 25½ inch. from the centre of the cyclotron (corresponding to nominal H_2^+ ion energies of 9 and 18 MeV respectively) and in argon and air at 3-4 $\times 10^{-5}$ mm. Hg and 10^{-4} mm. Hg. The results are shown in a graph and generally agree with the values calculated, using Salpeter's formula quoted in the article. The ratio of the measured cross sections of air and argon were within 5% of the theoretical values. At a pressure of 4×10^{-5} mm. Hg 70% of the H_2^+ ions reaching an energy of 1 MeV were lost by dissociation. If the pressure was reduced to 10^{-6} mm. Hg which is still possible in large vacuum systems, the loss would be reduced to 3%. But in the case of a synchro-cyclotron the vacuum required for efficient acceleration of H_2^+ ions would have to be better than is obtainable with present techniques. At a pressure of 2×10^{-7} mm. Hg in a typical 200 MeV synchro-cyclotron with a total path length of about 100 km 50% of the H_2^+ ions would be lost by dissociation.

Sommaire : Discussion de la dissociation des ions d'hydrogène moléculaires dans le cyclotron.

Article by K. E. Effat *Proc. Phys. Soc.* **65A**, June 1952 433-436

A Summary of the Present Status of Flow and Heat Transfer in Rarefied Gases

211/I

See Abstract No. : 178/I

Nozzle Characteristics in High Vacuum Flow—Rarefied Gas Dynamics

212/I

See Abstract No. : 180/I

Measurement of the Thermal Accommodation Coefficients of Gases

213/I

United Kingdom. Widely different values of the accommodation coefficients of inert gases at metal surfaces have been reported in the recent literature but reasonably concordant results have generally been obtained by different workers when the flashing procedure of Roberts was used. This technique was therefore critically examined by Bremner who concluded that the low values of Roberts were in error because the original thermal steady state was not attained. If this criticism is valid, then a re-assessment of Roberts conclusions in the application of them to the chemisorption of gases on metals is necessary. The techniques employed by Roberts and Bremner have therefore been investigated and have shown that the conclusions given by Roberts are substantially correct.

(*Science Abstracts*)

Sommaire : Le procédé dit ' le flashing ' employé par Robert pour la détermination du coéfficient d'accomodement de gaz inertes aux surfaces métalliques a été critiqué. Les auteurs ont étudiés la méthode et démontrent que les conclusions tirées par Robert sont substantiellement correctes.

Article by A. E. J. Eggleton, F. C. Tompkins & D. W. B. Wanford *Proc. Roy. Soc.* **213A**, 24.6.52 266-273

16 — CHEMISTRY — 16

Evaluation of Several Types of Replicas

214/I

See Abstract No. : 142/II

Russell Effect on Evaporated Metal Films

215/I

United Kingdom. Russell's discovery (in 1897) that freshly abraded metal surfaces produce an image on photographic plates due to the evolution of hydrogen peroxide has been re-confirmed in experiments with certain metals such as aluminium, magnesium, nickel or zinc which were cut under water and produced measurable quantities of hydrogen peroxide in the process. Extending the investigation to freshly evaporated metal films it was found that they, too, produced an image on photographic plates when exposed to oxygen and water vapour. An aluminium film deposited on a glass plate was moved into a light-tight box situated in the evacuated chamber and holding a photographic plate. Then, air containing 1.8% (volume) of water vapour was bled in. After 24 hours the plate was removed, developed in the usual way and the optical density measured. Plottings of the image density against pressure indicate that the image density increased with the

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pressure up to approximately 10 mm. Hg but at higher values became independent of pressure. A tentative explanation of the causes of the effect is added.

Sommaire : On a découvert en 1897 que la surface d'un métal fraîchement abrasé produit une image sur une plaque photographique. Des phénomènes simulaires ont été découverts sur des films metalliques fraichement déposés quand ceux-ci furent exposés à l'action de l'oxygène et la vapeur d'eau.

Letter by L. Grunberg & K. H. R. Wright *Nature* 170, 13.9.1952 456-457

Use of the Hollow Cathode and the Vacuum Furnace in Spectroanalysis

216/I

Belgium. A thin-walled hollow cathode of graphite, heated to 2,500° by a discharge through a rare gas, such as A, proves to be an excellent source for the spectrochemical analysis of very small samples. The lines of the alloying elements and impurities appear in succession with increase in temperature and pressure of the inert gas. This source is of use also in studying occluded gases, such as 0, which unites with the C of the cathode and emits CO bands. Similar results for metals and alloys can be obtained with a graphite-tube vacuum furnace which can be heated up to 2,500°. The occluded gases are studied by means of their absorption spectra.

(*Chemical Abstracts*)

Sommaire : Four à vide à arc contenant une cathode creuse en graphite développée spécialement pour l'analyse spectrochimique de très petits échantillons.

Article by B. Rosen & I. Ottelet *Colloque Int. Spectrograph. Strasbourg* Oct. 1950 155-67

The Effect of Sodium Bicarbonate on the Combustion of Cellulosic Materials

217/I

United Kingdom. The article reports on an investigation into the mechanisms of flame - retardation or smouldering and aims at establishing the best method of applying sodium bicarbonate for the purpose. A pyrolysis of various sodium bicarbonate-treated celluloses was carried out using a hard wood pulp with an alpha - cellulose content of 90% and a moisture content of 7%. The experimental equipment consisted essentially of a Pyrex tube, 1½ inch. diameter, the upper half of which contained the cellulose mixture and the lower half, separated by a gauze disc, fibrous glass to serve as a condensing surface for high molecular weight tar fractions. A receptacle for aqueous distillate and an acid bubbler were connected to the evacuation line for the determination of the low molecular weight tar fractions. The required heat, 300 to 600°C, was supplied by a furnace and the progress of the pyrolysis followed by the recordings of a thermocouple situated in the base of the pyrex tube. The quantities of tar, char and moisture obtained during a distillation run taking about 2½ hours were recorded in % of the original weight of the charge as shown in Table I partly reproduced below.

Pyrolysis Reactions

(*Hardwood Pulp with a Moisture Content of* 7%)

Added Chemical	*% Added (wt./wt. Basis)*	*% Tar*	*% Char*	*% Aqueous Dist.*	*Ratio Tar/Char*
None		44.5	19.8	27 8	2.24
$NaHCO_3$ powder	1.2	36.0	24.0		1.50
	16.4	19.5	37.0		0.52
	30.4	18.5	36.2		0.51
	67.7	19.5	38.5		0.51
$NaHCO_3$ impregnation	1.2	28.0	34.0	28.5	0.82
	6.4	11.0	49.0	29.3	0.22
	17.0	10.2	51.3	30.0	0.20

Impregnation had a better effect than admixture in the form of powder. Plottings of the pressure variations in the system during the reaction against time showed two pressure peaks in the case of untreated cellulose, the first indicating distillation of hygroscopic water and the second the main reaction. In the interval the pressure decreased due to the production of vapour products. In the case of impregnated cellulose the same double peak appeared but the main reaction was greatly retarded. In the case of admixture in the form of powder three peaks were shown, the first due to distillation of hygroscopic water, the second symptomatic for an abnormal production of primary tar occurring at temperatures below normal reaction temperatures and the third indicating final degradation and producing a brown tar and a considerable amount of gas. Cellulose plus powder increased the gaseous product appreciably. The solid and aqueous combustion products of cellulose material with sodium bicarbonate admixed either way were examined in a separate series of control experiments. The apparatus used consisted of a Pyrex combustion tube divided into two sections. The upper section contained the charge and the lower section means for the collection of the tarry-distillate. The aqueous distillate was collected in a trap. Air was drawn through the tube maintaining the system at a partial vacuum. The rate of flow could be stabilised. As a first step the combustion tube was heated up to 200°C, the vacuum pump started and the flow adjusted to the set standard. Subsequently the temperature was raised by 10°C every minute until ignition occurred. Compared with the results from untreated cellulose, impregnation reduced the amount of tar produced by 17% and increased the amount of char produced by 13%. For admixed powder the figures were 1% and 2% respectively. It is concluded that

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impregnation ensures more intimate contact with the cellulose fibres than admixture in powder form. The method of application decides whether flame retardation or low temperature smouldering is produced. Both phenomena are expressions of the same reaction mechanism.

Sommaire : Recherche par méthode du vide sur les propriétés flamifuges de bicarbonate de soude mélangé a des matières cellulosiques.

Article by J. E. Dolan *Chem. & Industr.* 26.4.1952 368-371

Production of Ammonium Nitrate by Continuous Vacuum Crystallisation 218/I

See Abstract No. : 214/III

Homotransplantation of Tracheal Segments Preserved by Lyophilisation. An Experimental Study 219/I

United States. In order to determine whether freeze-dried tracheal segments can be used as homografts in animals, tracheas taken from dogs which had been sacrificed were freeze-dried at a temperature of –15 to –25°C, kept in the apparatus from 72 to 96 hours at a pressure of 0.001mm. Hg and then transferred to sealed jars for storage at room temperature. The trachea sections used ranged from 2 to 10 cm. After freeze drying the tracheal segments looked like rigid tubes fully devitalised. But prior to use all segments were hydrated with physiological saline and as a result they took on normal appearance again. The outcome of the experiments is shown in a table reproduced below in abbreviated form.

Table I. Results of Transplantation of Lyophilised Tracheal Homografts in Dogs

Dog. No.	*Length of Time Graft Preserved (Days)*	*Length of Life Postoperatively (Days)*	*Cause of Death*	*Fate of Graft*
4– 1–50	10	8	Sacrificed	Excellent
4–10–50	30	10	Pneumonia	Necrosis of Graft
4–20–50	90	90	Sacrificed	Excellent
4–25–50	120	90	Sacrificed	Excellent
5– 1–50	8	120	Sacrificed	Excellent

Healing occurred in all cases except one. It was found that these transplants acted in the same manner as fresh homografts. A slight necrosis occurred in the wall of the graft during the first two weeks but this did not interfere with the ultimate healing of the graft.

Sommaire : Description d'une éxperience faite en vue de déterminer s'il etait faisable de lyophiliser des segments de trachée en vue de l'homo-transplantation.

Article by A. G. Marrangoni *J. Thoracic Surg. St. Louis* 21, April 1951 398-401

A New Method for Gettering and Preserving Organ Powders Destined for Therapeutics by Using Low Temperature, Desiccation and Vacuum 220/I

See Abstract No. : 78/IV

Experiments with an Apparatus for Lyophile Desiccation of Micro-Organisms 221/I

See Abstract No. : 76/IV

The Molecular Distillation of Fats and Oils 222/I

See Abstract No. : 205/III

17 — METALLURGY — 17

The Protective Properties of the Natural Oxide Film on Aluminium 223/I

United Kingdom. Aluminium is a highly reactive metal and its known resistance to atmospheric corrosion or immersed corrosion is due to the presence of a thin, adherent and protective oxide film which forms spontaneously on exposure to air or liquids containing salt, oxygen or water. Hass has shown that a film of 15-20 Å forms immediately on exposure to air. Subsequently the growth slows down and after one month ceases completely. The rate of growth is influenced by various factors such as temperature, surface conditions, etc. and reaches a maximum in the presence of air saturated with water. Films formed at high temperatures are thicker than those formed at room temperatures. Films formed on rough surfaces are thicker than those formed on smooth surfaces. It is not clear whether the resistance of the film to further oxidation is due to its

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resistance to the passage of electrons or of ions. Mott suggests that electrons can pass the film easily, ions only in the presence of a strong field. If the oxygen supply to the metal surface is restricted corrosion may occur if moisture is present. Rakowski showed that aluminium foil tends to corrode in a moist atmosphere if wound in a coil but not when freely exposed. Aluminium foil of the type used in the food industry is liable to give trouble if it shows pinholes. The author proceeds to discuss the conditions applicable when the aluminium surface is immersed in an aqueous solution. Here, the concentration of oxygen is restricted due to its low solubility and the amount of oxygen reaching the surface does not suffice to maintain a film and prevent corrosion. The natural oxide film is very thin, in fact invisible. It can be artificially thickened by chemical oxidation or by anodising. The latter method facilitates the production of films with a wider range of properties than the former. Anodic films are corrosion-resistant in neutral solutions, but are liable to attack by acid or alkaline solutions. The porosity of the films can be reduced or eliminated by treatment with boiling water. Anodising followed by lacquering is an effective means of protecting the aluminium surface from corrosion by food products. The mechanism of corrosion as far as it is explored to date is discussed. Finally details are given from a study undertaken by the low Temperature Research Station, Great Britain, defining and assessing the various factors influencing the corrosion of aluminium by citric acid and citric acid-chloride solutions.

Sommaire : Recherches sur la croissance et les propriétés de films d'oxydes naturels, recouvrants une surface d'aluminium.

Article by J. M. Bryan *Chem & Industr.* 23.8.52 820-822

O

The Formation of Refractory Coatings by Vapour Deposition Processes

224/I

United States. The present state of the vapour deposition technique is reviewed (see also Abstract No. 170/I). The technique is used for the deposition of coatings of refractory materials. The operational temperature is far below the melting point of the material used for coating and in a range where the respective vapour pressure is negligible. Most vapour deposition processes can be carried out either in a vacuum or at atmospheric pressure. There are two groups of processes : (1) chemical reduction or displacement at the surface to be coated and (2) thermal decomposition at the surface to be coated. A general description of the procedures is given and a sketch shown of a continuous-flow wire coating apparatus based on this method. The following is a list of suitable materials:

Metals

Beryllium,	Aluminium,	Titanium,	Zirconium,
Hafnium,	Thorium,	Vanadium,	Niobium,
Tantalum,	Chromium,	Molybdenum,	Tungsten,
Uranium,	Rhenium,	Iron,	Nickel,
Ruthenium,	Rhodium,	Osmium,	Iridium,
Platinum,			

Alloys of

Tantalum and Titanium	Tantalum and Zirconium,	Tantalum and
Chromium and Molybdenum	Chromium and Iron	Niobium

Metalloids

Boron	Carbon	Silicon

Compounds

Carbides of			
Boron,	Silicon,	Titanium,	Zirconium,
Hafnium,	Vanadium,	Niobium,	Tantalum,
Chromium,	Molybdenum	Tungsten	
Bitrides of			
Boron,	Titanium,	Zirconium,	Hafnium,
Vanadium,	Viobium,	Tantalum	
Silicides of			
Titanium,	Zirconium,	Niobium,	Tantalum,
Chromium,	Molydbenum,	Tungsten	
Borides of			
Aluminium,	Silicon,	Titanium,	Zirconium,
Hafnium,	Vanadium,	Niobium,	Tantalum,
Chromium,	Molybdenum,	Tungsten	
Oxides of			
Aluminium,	Silicon,	Titanium,	Zirconium,
Chromium			

O

A typical vacuum deposition process is the following :

$$TiI_4 \xrightarrow[\textit{in vacuo}]{1200^\circ\text{–}1400^\circ\text{C}} Ti + 2I_2$$

The higher the concentrations and pressures the finer are the crystalline structures of the deposits. The adhesion depends on the deposition temperature, the purity of the reactants, the cleanliness and the chemical properties of the surface to be coated. At high temperatures the less stable compounds tend to be reduced or

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decomposed at a considerable distance from the surface to be coated. To overcome this one can lower the temperature of the surface or reduce the pressure in the chamber. Deposition rates vary. The hydrogen reduction method is faster than the thermal decomposition method. The efficiency of the process, as measured by the ratio of material deposited to material supplied as volatilised compound, varies greatly in each individual case. The thermal decomposition processes carried out in a vacuum are near 100%. The article ends with a detailed discussion of the actual and potential applications of the process.

Sommaire : Revue de l'état actuel des développements concernants le dépôt de revêtements réfractaires à partir de la phase gaseuze.

Article by C. Powell & I. E. Campbell *J. Chem. Educ.* **29**, April 1952 181-186

Ion Producing Mechanism

225/I

United States. An ion generator is described of the type which generates ions by bombarding a solid body with electrons. It consists of a glass cylinder fitted with metal plates at both ends, appropriately sealed to facilitate evacuation. The bottom plate supports an alundum crucible which holds the material to be ionised, *e.g.* uranium. A tungsten wire sealed through the chamber wall is in contact with the uranium. Near the top of the vessel is a filament acting as the electron source and between the filament and the top end plate is a grid of wire mesh. The filament and the uranium form a (battery driven) circuit where the filament is the cathode and the metal the anode. The upper end plate is rendered negative with respect to the filament and the grid. The latter is of the same potential as the filament. After evacuation the filament circuit is closed and a stream of electrons strikes the uranium in the crucible. As a result the uranium is vaporised. The rising vapours are bombarded by the electron stream and set up an arc discharge. The positive ions of the arc plasma are attracted by the grid and, after passing the same, are accelerated towards the end plate, causing a current to flow through the ammeter connected with the end plate. This gives an indication of the number of ions removed from the plasma. The apparatus is recommended for use in conjunction with a calutron or any other device where ions of uranium or any other metal are required.

Sommaire : Description d'un générateur d'ions construit pour extraire des ions (d'uranium, par example) en vue de leur emploi dans des accélérateurs.

J. G. Backus U.S. Atomic En. Comm. *U.S. Pat.* 2,600,151

The Use of a Tungsten Filament as an Evaporation Source for Silver

226/I

See Abstract No. : 179/III

Evaporation of Silver from Electroplated Heating Elements

227/I

See Abstract No. : 178/III

Vacuum Impregnation of Castings

228/I

See Abstract No. : 195/III

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

Working Principle of the Vacuum Pumps 124/II

See Abstract No. : 177/I

A High Vacuum High Speed Ion Pump 125/II

United States. Ionisation has never been used deliberately for the evacuation of a system on a scale comparable to that of a commercial diffusion pump. In this report the authors describe the construction and performance of an ion pump specially developed for the purpose. The pump consists of a tube, also serving as the anode, with an outlet in the centre for connection with the vessel to be pumped and a hot cathode at one end and a cold cathode at the other. The discharge in the tube is collimated at both ends owing to the provision of an axial magnetic field. Electrons emitted from the hot cathode are accelerated into the discharge and travel through the tube until they reach the opposite end, where they are reflected by the cold cathode. The electrons oscillating in this manner lose much of their energy by ionisation before they reach the anode walls. As a result a positive space charge develops which moves the gas molecules, ionised on entering the discharge in the centre section, to the cathode. As this process continues the gas pressure in the system is reduced until an equilibrium is reached. On reaching the cathode area the ions either combine with the cathode material or become neutral again. In the latter case they may attempt to travel back but only to be ionised again and returned to the cathode. As long as the gas flow does not exceed 0.02 c.c. per second NTP no forevacuum system is needed. The gas ' pumped ' is disposed of by adsorption and chemical binding. But at very low pressures the ion supply in the centre region of the pump is insufficient to maintain the discharge. To overcome this, gas is leaked into both ends of the tube to raise the pressure in the cathode region above 3×10^{-4} mm. Hg. The long anode restrictions prevent the high pressure regions from spreading to the interior of the tube. Operational data of an ion pump is given as follows :

Pumping Speed	3,000-7,000 l./sec.
Base Pressure	0.8×10^{-6} to 5×10^{-6} mm. Hg
Arc Voltage	400-300 V.
Arc Current	20-10 A.
Cathode	Radiantly Heated Tungsten Cathode. Power Consumed 4.5 kW.
Magnet Power	Side Magnets 20 kW. Centre Helix 12 kW.

It has a power input of 42kW and has been used for the pumping down of a 48,000 litre-tank from 10^{-2} mm. Hg to 5×10^{-6} mm. Hg. The pump can be run continuously for about 2-4 weeks.

Sommaire : Premier rapport détaillé sur la construction d'une pompe à ionisation à grande vitesse, développée à Berkeley.

Report by J. S. Foster, Jr. E. O. Lawrence & E. J. Lofgren *Radiation Lab. Univ. Calif. Rep. No.* UCRL-1930 27.8.52

Method for the Production of a Vacuum 126/II

Germany. In roll coating processes where continuous bands are metallised by evaporation a major difficulty is presented by the problem of introducing the band into the vacuum chamber. Where the rolls are positioned outside the evacuated coating chamber the slots, required for the introduction of the band, admit air at the same time. In the past, plant of this description was fitted with forevacuum chambers which were continuously pumped to counteract the influx of air through the slots. In the present invention, too, the band passes through at least 2 chambers before it reaches the coating chamber but no pumps are required for their evacuation. The first, receiving the band from atmosphere, is filled with a condensable gas such as water vapour at a pressure slightly above atmosphere, to stop the air from penetrating through the slot. The second is fitted with refrigeration equipment causing all gas reaching that chamber to condense. In this way one can obtain a satisfactory vacuum as air or permanent gases are absent. A plant is shown in a sketch consisting of three chambers on either side of the coating chamber, one filled with water vapour and two fitted with refrigerating means to condense the gases penetrating from one chamber to the next at successively lower temperatures.

Sommaire : Il est proposé de produire le vide en condensant de la vapeur d'eau introduit pour l'élimination de l'air dans une chambre multiple construite pour la métallisation en rouleaux.

W. Winter & R. Bosch G.m.b.H. *German Pat.* 752,050

On the Assessment of Back-Streaming in Vacuum Systems 127/II

United Kingdom. A method is described of testing the efficiency of cold traps in vacuum systems. Good performance of cold traps is of particular importance where, as in a coating plant for instance, the substrate is kept at a temperature lower than that of its surroundings. Oil back-streaming from the pump will contaminate the substrate. An Adam trough is used in this method, filled with water, the surface of which is sprinkled with fine talc powder. When the substrate is dipped into the water, the oil spreads across the surface and forms a monomolecular film. The oil pushes the powder back and the area finally covered is well defined by a sharp boundary. A calibration method is described which facilitates an assessment of the amount of oil deposited. The results of two experiments are shown in graphs.

Sommaire : Méthode de détermination de la rédiffusion de l'huile d'une pompe à diffusion.

Letter by S. G. Bradley G. K. T. Conn & G. K. Eaton *Vacuum* **2**, Oct. 1952, 387-388

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

21 — GAUGES — 21

A Precision Differential Manometer 128/II

United States. For a study of the flow and heat transfer characteristics of low density gases a pressure measuring device was required which facilitated rapid and continuous measurements in the range of 0.05 mm. Hg to 20 mm. Hg. Other major stipulations were: consistent accuracy of readings, reasonably rapid response to pressure changes and elimination of the effects of surface tension so that readings taken in an increasing pressure cycle could be compared to those taken in a decreasing pressure cycle. The newly developed instrument consisted of two parallel lengths of 10 mm. diameter precision bore tubing fitted with 'O'-ring seals into a baseplate and upper metal block. The upper block contained the pressure connecting channels, valves and by-pass connections and the baseplate contained the drain port. The meniscus viewing system gave a magnification of 4 × and was mounted on lead screws joined to a gear train controlled by a hand wheel. One turn of the wheel raised or lowered the system by 0.05 inch. The motion of each lead screw was communicated to a precision differential which indicated the addition or subtraction directly on a counter. Methodic experiments to test the accuracy of the instrument covering both increasing and decreasing pressure cycles established that the mean deviation from calibration values could be kept within ± 0.7 micron Hg. Readings could be reproduced by different observers with a precision of 1 micron Hg. N-butyl phthalate was used as the fluid.

Article by G. J. Maslach *Rev. Sci. Instrum.* **23**, July 1952, 367-369

Sommaire : Description d'un nouveau manomètre différentiel de précision et de ses performances.

Shunted Thermocouple Vacuum Gauge 129/II

Australia. It is proposed to improve the sensitivity of the thermocouple gauge by adding a shunt to the heater circuit inside the gauge. In the conventional design the heater current (the voltage remaining constant) is comparatively small at low pressures and the sensitivity is correspondingly reduced. To overcome this a barretter is normally employed which keeps the heater current at a constant value. However, the change in the resistance of a conductor with temperature could be used for increasing the sensitivity of the gauge. If it is assumed that two resistances are connected in parallel, R_1 with a high temperature coefficient and R_2 with a negligibly small temperature coefficient, then the relation between I, the total current (maintained constant) and the variation of I_2 (through R_2) is given by $\triangle I_2 = I R_2 \triangle R_1/(R_1+R_2)^2 = I R_1 R_2 \alpha \triangle t/(R_1+R_2)^2$ where $\triangle t$ is the increase in temperature. The proposed circuit is shown in a sketch, R_2 the thermocouple heater, is made of Nichrome and Eureka wire and R_1, the shunt, is made from molybdenum wire. The pressure is inversely proportional to the temperature and resistance of the shunt. Thus, at low pressures the current will be greater. But the increase of the heater current has practical limitations. It is essential that the values of both resistances, heater and shunt, are such that their temperatures are approximately the same. Design details and the calibration curve of a gauge incorporating a shunt are given.

Note by N. A. Florescu *J. Sci. Instrum.* **29**, Sept. 1952 298

Sommaire : Il est suggéré d'améliorer la sensitivité de jauges à thermocouple par l'addition d'un shunt dans le circuit de chauffage.

Hot Wire Manometers 130/II

Sweden. The determination of heat conductivity of gases by a 0.01 mm. diameter tungsten wire extended in a 10-20mm. diameter glass tube, the wire forming one arm of a Wheatstone bridge, is described and Knudsen's formulae for the molecular conductivity, valid for pressures smaller than 10^{-3} Torr, and for conductivity at high pressures are discussed. Voege's thermo-cross and Pirani's resistance manometers are described. The volt/ampere characteristics for pressures of 0-10 Torr are given and the working lines for constant current, constant voltage, constant power and constant temperature are shown. A complete circuit diagram for a constant temperature manometer is given. Calibration curves for the various types are shown.
(*Science Abstracts.*)

Article by G. Von Dardel & H. Von Ubisch *Tekn. Tidskr.*, **82**, 4.3.52 203-8

Sommaire : La construction et les performances de manomètres à fil chaud dans la domaine allant jusqu'à 10 mm. Hg ont été étudiés.

Hot Wire Manometers for Chemical Applications 131/II

Sweden. The conventional hot wire or Pirani-type manometer covers a pressure range from 10^{-4} to 10^{-1}mm. Hg. The Pirani-type manometer would have many useful applications in chemical work, if the upper limit of the present range could be extended to the order of pressures where liquid level manometers can be employed. The author investigates the possibilities of designing such a gauge and discusses the working principle of the gauge and the theory of heat conduction in gases as far as it affects the operation of the gauge. The basic formulae governing the operation of the gauge in gases of molecular, transmolecular or molar state are given. An 0.01 mm. wire should be used in the case of molecular conductivity and a ribbon of 10 mm. width in the case of molar conductivity. The accuracy of a gas analysis by means of thermal conductivity depends on the heat conduction properties of the gas mixture in question, *i.e.* the degree of discrimination possible between the constituents, and good control of operating conditions (*e.g.* ambient temperatures). Of the three working principles of the gauge, the one providing for continuous adjustment of the heater current (*e.g.* by electronic feedback) to ensure constant wire temperature, offers the greatest scope for the development of a gauge which covers a wide range of pressures up to the level where heat conduction is no longer affected by pressure. The

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tungsten wire for such a gauge should have a diameter of 0.01 mm. and a cold resistance of 7.6 Ω per cm. For an ' infinite length ' of this wire the heater current is 1.5 mA at pressure zero, 6.3 mA at a pressure of 0.1mm. Hg and 45 mA at a pressure equal or higher than 100 mm. Hg, if the gas is air. The temperature of the wire should not exceed 150°C. In the last part of the article the author gives a full description of the design of a gauge for pressure measurements up to 100 mm. Hg, operating with electronic feedback. Block diagrams of the general assembly and the feedback circuit are shown. The gauge is self-stabilising against fluctuations of line voltage. This feedback wide-range manometer proves particularly useful in work with corrosive compounds. When dealing with condensable vapours it may serve as a convenient substitute for the alphatron gauge. It can be employed with advantage in leak detection as it remains highly sensitive to tracer gases, such as hydrogen and coal gas, at all pressures up to atmospheric.

Sommaire : Les facteurs régissant la construction de manomètres à fil chaud du type Pirani, afin de les rendre utiles pour les procédés chimiques, sont discutés et les détails d'une construction développée spécialement en vue de la mesure dans la région des 10 mm. Hg, sont donnés.

Article by H. Von Ubisch *Analyt. Chem.* **24**, June 1952, 931-938

The High Temperature Pirani Gauge

132/II

United Kingdom. The author refers to work published recently by B. G. Dickens and H. Von Ubisch who extended the working range of the Pirani gauge to a minimum top limit of 30 mm. Hg. The design of these gauges is based on the constant temperature bridge principle, but this principle is not popular because the supply voltage must be adjusted before each reading. Normally the constant voltage bridge principle is used. The present paper gives a theoretical analysis of operating conditions in the pressure range up to 2,500 μ Hg and an assessment of the effects of wire temperature, type of bridge circuit and fluctuations of ambient temperature and supply voltage on the final value of the upper limit of the operational range. Formulae are developed defining thermal equilibrium in the wire for the three cases : (*a*) Constant temperature bridge, (*b*) constant voltage bridge and (*c*) constant current bridge. Experiments were carried out which confirmed the results of the theoretical considerations, and in addition threw some light on the performance of tungsten and platinum wire as detector elements. The author comes to the following conclusions : Unless ambient temperature fluctuations can be eliminated the range of the gauge can only be extended up to and above 1mm. Hg by increasing the wire temperature above the normally accepted 400°K. But both tungsten and platinum wires give trouble at that temperature, tungsten because of oxidation and platinum because of a change of surface conditions. For a range exceeding 100 μ Hg the ratio of fixed resistance : resistance of the measuring arm should be 1 : 5 to 1 : 7.5. A pronounced shift of the zero reading has been observed in the pressure region of 10-20 μ Hg. Recommendations are added for the design of a gauge to be operated at various pressure levels.

Sommaire : Recherches complètes sur les préceptes de construction et les performances d'une jauge Pirani opérante dans le domaine des mm. de Hg.

Article by J. H. Leck *J. Sci. Instrum.* **29**, Aug. 1952, 258-263

Pressure Measuring Device

133/II

United States. Normally the Philips-type gauge can be used in pressure regions up to 1 micron Hg only. The design described here can be used for the measurement of pressures up to 20 micron Hg. It consists essentially of an annular anode and an annular cathode placed in a plane perpendicular to the magnetic field. The cathode is slightly larger than the disc-like anode, and is C-shaped in section, the open space of the C facing the outer edge of the disc. All points in the electric discharge, except those lying exactly in the plane of symmetry through the electrodes are subjected to an electric field with one component parallel to the magnetic field and another component perpendicular to the magnetic field. The parallel component accelerates electrons in the direction of the plane of symmetry of the electrode. The electrons travel through that plane until they are stopped and reversed on the other side at a point of the same potential as their point of departure, *i.e.*, the electrons are caused to oscillate across the plane of symmetry. At the same time the other component of the electric field forces the electrons in a circumferential direction around the annular path of the electric discharge. As a result the electrons oscillate for an unusually long period, and ionise a sufficient number of atoms of the gas present to establish a glow discharge. The gauge can be operated with a.c. or d.c. Two embodiments of the new design principle are described.

Sommaire : La construction d'une jauge Philips, qui permet d'opérer avec précision jusqu'à des pressions de 20 microns Hg, est décrite.

W. A. Arnold & U.S. Atomic En. Comm. *U.S. Pat.* 2,577,066

Ionisation Gauge

134/II

United States. Conventional ionisation gauges work on the principle of temperature-limited emission. The emission current is usually maintained at a constant level by suitable adjustment of the filament current either manually or with regulating circuits. The measurement of the positive ion current is then a measure of the pressure. The patent describes the use of a tetrode or triode ionisation gauge in which the emission is not temperature-limited but controlled by the potential on the control grid of the tetrode or the grid of the ionisation gauge. The screen grid of the tetrode and the plate of the triode ionisation gauge are at a positive potential and receive the electron current, while the plate of the tetrode and the grid of the triode are at a sufficiently negative potential to collect the positive ions. The patent covers various circuits by which the emission is automatically regulated in tetrode or triode ionisation gauges working under these conditions.

Sommaire : Description d'une jauge à ionisation tetrode où triode où l'émission n'est pas limitée par la température mais contrôlée par le potentiel de la grille re. réglage de la jauge ou de la tetrode.

K. M. Simpson, W. R. Baker, Q. A. Kerns & U.S. Atomic En. Comm. *U.S. Pat.* 2,595,611

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

A Demountable Tetrode Ionisation Gauge

135/II

United Kingdom. A demountable tetrode ionisation gauge with a tungsten filament that can readily be replaced is described. The additional grid in the gauge provides a simple means of stabilising the electron current. The measured value for the stabilisation agrees with the value calculated from the measured current-voltage characteristics of the gauge and is comparable with the stabilisation achieved using the more complex circuits needed to stabilise the emission in a triode ionisation gauge. The disadvantage of the gauge is that due to the heat of the filament much gas comes from the vacuum seal and any nearby metal walls, unless they are cooled. Tetrode gauges mounted on pinches inside glass envelopes are satisfactory from this point of view. (*Author*)

Sommaire : Jauge à ionisation à tétrode démontable et filament de tungstène.

Report by J. E. Johnston *A.E.R.E. Rep. No.* G/R 480, Jan. 1950

An Investigation of a Metal Knudsen Manometer

136/II

Denmark. A Knudsen manometer constructed from metal is briefly described. Details are given of the calibration procedure covering the pressure range 10^{-3} to 10^{-4}mm. Hg. Finally degassing effects are discussed.

Sommaire : Performances d'une jauge de Knudsen métallique.

Letter by H. G. Jensen *Vacuum*, **2**, Oct. 1952, 386-387

22 — MEASURING PLANT (VACUUM) — 22

Free Molecular Flow in the Sample Inlet to the Mass Spectrometer

137/II

See Abstract No. : 72/IV

X-Ray Shadow Microscopy

138/II

United Kingdom. The general scope of the method, its possibilities and limitations, are discussed. (See also Abstract No. 139/II Vol. I). In the camera described tungsten foil 1 μ thick is used as a target and the object is placed as near as possible to it, in air. The electron beam is focussed by means of electron lenses to a point not exceeding 1 μ diameter. The recording emulsion should be close to the source for the sake of a short exposure time, but on the other hand not too close, so that image details remain larger than grain size. With an object-target distance of less than 1 mm. the camera gives an initial magnification of 100 ×. A micrograph is shown of a silver grid 1500 meshes per inch consisting of bars 2 μ and 3 μ wide. The initial magnification is 100 ×, and the total magnification 700 ×. The resolution obtained is estimated at about 0.5 μ. This grid was used as a standard when examining biological specimens which are particularly well suited for a study by this method due to their differential absorption of X-rays. A micrograph of the drosophila melanogaster and another of the head alone of the same insect are shown. Some degree of drying is necessary to give the required contrast. For that reason the specimens shown had been freeze-dried. This method appeared to produce the least distortion. In order to follow internal processes one can use living specimens which are temporarily anaesthetised but the resolution is not so good. The greatest problem with regard to future developments is the electron intensity available. It is known that the energy dissipation from a focal spot becomes relatively more rapid as its diameter is reduced. On the other hand, the tungsten filament (electron source) can only provide a limited electron intensity. In the arrangement shown practical exposure times range from 1-10 minutes.

Sommaire : Construction et utilisation d'un microscope d'ombres à rayons X.

Article by V. E. Cosslett & W. C. Nixon *Nature*, **170**, 3.9.52 436-438

Practical Applications of Electron Diffraction

139/II

United States. The electron diffraction camera consists essentially of an electron gun, a tungsten filament, which produces a parallel beam of electrons. The electrons are accelerated and directed by a magnetic focussing coil on to a small spot of a fluorescent screen or photographic plate. An auxiliary fluorescent screen is available for visual examination. There are two methods of obtaining diffraction patterns, by transmission and by reflection. As little as 5 micro-micrograms of material, spread in layers as thin as 15 Å, are required as a sample for a chemical analysis. The camera is also used for the determination of crystal size and orientation of a material. There are many cases where X-ray diffraction methods yield comparable results. That technique has the advantage that the specimen need not be kept in an evacuated space. But the electron diffraction method has the advantage that the exposure times required are appreciably shorter, *i.e.*, 0.1 to 2 minutes as compared with 1 to 10 hours. In addition, the normally used electron voltage (appr. 50 kV) yields wavelengths about 1/20 of those used in X-ray diffraction. Thus electrons are diffracted by shorter spacings and allow the estimation of crystals smaller in size than those covered by the X-ray diffraction method. The penetrating power of 50 kV electrons does not exceed 1,000 Å units but the same voltage causes X-rays to penetrate a millimeter or more. Thus, electrons are ordinarily used to give information on the surface structure of a sample, whilst X-rays are used for giving information on the body structure. The potential variety of electron diffraction applications is demonstrated by a number of examples. In one case, for instance the crystal habit of a thin evaporated layer of germanium was to be investigated. This element is known to change to a new crystal arrangement when heated to about 350°C. In order to determine the initial structure

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of the deposits the samples were first examined by X-ray diffraction, but found to be too thin for satisfactory observation by this method. On the other hand they were too thick for examination in the electron microscope and the individual crystals too small for the ordinary light microscope. By applying the electron diffraction method it could be proved that the original film was of normal structure. The individual crystals had a size of no more than 15 Å in diameter. But, after heating the sample to 350°C *in vacuo*, transformation took place as it does with the bulk material. In conclusion it is stated that electron diffraction methods are particularly suitable for the discovery of contamination and corrosion products.

Sommaire : Rapport sur les méthodes et utilisations des études de diffraction électronique.

Article by S. P. Newberry & E. I. Alessandrini *Instruments*, **25**, July 1952, 907-912

Electron Diffraction in Some Cubical Crystals 140/II

See Abstract No. : 209/I

Electron Diffraction in Paper Examination 141/II

United Kingdom. Electron diffraction methods facilitate the examination of paper surfaces with an accuracy unobtainable with other equipment. The electron diffraction camera is used for the testing of glazed paper surfaces produced by the Fourdrinier machine, by plate glazing or by super callendering. One can thus determine the depth of the glaze which is an important factor controlling the reproduction quality of fine screen half-tone illustrations. It is also used for the assessment of the structure and depth of various coatings on paper. New materials and new binders have recently been introduced for the purpose of coating paper, for instance, titanium calcium pigment, vacuum deposited metal coatings, and hydroxyethyl cellulose as a binder. These changes call for refined testing methods to ascertain distribution and density of the finish. Electron diffraction methods can help in determining the fibre structure which governs the strength of the paper and as the fibre structure of a finished paper can be varied by adjusting the production accordingly electron diffraction can be instrumental in obtaining the desired quality. Details are given of the construction of a diffraction camera and its auxiliary vacuum equipment. The procedure of interpreting and evaluating diffraction patterns is explained. In the majority of cases interpretation is effected by reference to comparison patterns obtained from standard substances, the lattice spacings of which are known.

Sommaire : Courte revue des techniques de diffraction électronique et de l'utilisation de cette technique pour l'essai de papier.

Article by A. E. Williams *World's Paper Trade Rev.* **138**, 7.8.52 421-423

Evaluation of Several Types of Replicas 142/II

United States. In a study concerned with photographic film base, single macromolecules of highly polymeric materials had to be examined in the electron microscope. In order to obtain true replicas of the surfaces investigated, all artefacts had to be excluded and substrates had to be used which exhibited a minimum of structure. This led to a separate investigation of the comparative merits of one-step silica and two-step polystyrine silica replicas reported in this article. Firstly, the quality of the two replication methods was investigated using replicas of the surface of gelatine cast on a cellulose ester photographic film base. Secondly the resolution properties of silica replicas were studied which were prepared by evaporating a hydrophilic layer of sodium chloride on to the gelatine film prior to the deposition of the silica. In the third section methods are described for the preparation of substrates of silicon dioxide or silicon monoxide showing a minimum of structure, and facilitating high resolution studies. All depositions were carried out by evaporation in a vacuum. The silicon dioxide and silicon monoxide substrates were deposited on the surface of freshly cleaved mica because it made separation under water possible. The deposits had a minimum thickness of 40 Å. A platinum-palladium alloy and chromium were employed for shadowing. By special treatment of the gelatine film two types of surfaces were obtained, classified as fine structure and rough structure. A shadowed specimen of each of these surfaces served as a standard for the assessment of the quality of the replicas. The standard rough structure film showed cavities 600-1,200 Å deep, and 600-3,000 Å wide. The standard fine structure film showed cavities 50-1,000 Å deep and 100-700 Å wide. The one-step silica replicas were generally in good agreement with the standards. Elevations of the order of 25 Å could still be determined. The polystyrine silica replicas, however, gave a relatively poor reproduction of the original surface. Narrow 'ridges' appeared separating the cavities. The use of an intermediate layer of sodium chloride would have helped separation under water but it was found that even at low magnification and with very thin layers sodium chloride would introduce its own grain. With regard to the substrates it was found that silicon dioxide substrates exhibited the least structure. At a film thickness of 200 Å silicon monoxide and silicon dioxide substrates showed a pronounced difference in structure but their electron scattering power (transmissivity) was the same. The authors state that they preferred the platinum-palladium alloy or uranium to chromium as a shadowing material. Chromium introduced some structure.

Sommaire : Revue des méthodes connues de réplication pour le microscope électronique et conseils concernant la production de répliques exposants un minimum de structure.

Article by J. J. Comer & F. A. Hamm *Analyt. Chem.* **24**, June 1952, 1006-1016

A New Device for Metallic Shadow Casting 143/II

See Abstract No. : 181/III

II — Vacuum Apparatus and Auxiliaries — II
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Abstract No. and References

26 — MATERIALS — 26

The Analysis of Heat Transfer through Thermal-Insulating Materials — 144/II

See Abstract No. : 202/I

On New Methods of Insulating Very Low Temperature Apparatus — 145/II

See Abstract No. : 202/I

Metal Coatings on Glass or Quartz Not Intended for Mirrors or Decoration — 146/II

See Abstract No. : 85/IV

Method of Forming a Vacuum-Tight Bond Between Ceramics and Metals — 147/II

Switzerland. Valves for ultra short waves are preferably made from burnt refractories such as steatite. Glass, the alternative material, does not withstand the high electric stress. An important process in the manufacture of these valves is the vacuum-tight bonding of the ceramic to metal. One of the methods known consists of metallising the ceramic and subsequently brazing the metal component on to the metallised surface. The metal layer is sprayed on or burnt in. This method is complicated and of little practical use. The new method proposed in this patent is simpler. It is based on the operation of pressing the metal and the ceramic together at an elevated temperature. This has been found to result in a vacuum-tight bond. The temperature and pressures used can be varied, but the lower limits of both are determined by the conditions under which the metal becomes plastic. Another important factor is the time allocated to the pressing operation. Four applications of the process are described. In one application copper covered with cupric oxide is heated until red hot, and then dipped into water. As a result the cupric oxide changes into cuprous oxide. Pressing is carried out in a nitrogen atmosphere. The temperature is 1,000°C and the pressure is 3,000 lb. sq. inch. After a treatment lasting a few seconds the combined material shows a higher tensile strength than the bare steatite.

Sommaire : Méthode consistant en l'application d'un recouvrement métallique sur de la céramique en comprimant un matériel sur l'autre en vue d'obtenir un joint résistant au vide.

F. Tank, & Ges. Foerd. Forsch. Eidg. Techn. Hoch. Zuerich *U.S. Pat.* 2,564,738

28 — APPARATUS COMPONENTS — 28

A Quick Release Seal for Vacuum Desiccators — 148/II

Abstract No. : 158/II

Shaft Seal for Vacuum Apparatus — 149/II

United States. Although there are a great many shaft seals available ready-made, the research worker has still to resort frequently to fabrication in order to satisfy his requirements. The authors describe the design of a shaft seal simple to fabricate and suitable for driving the stirrer of a pot still. It consists of a drill rod 10 inch. long, rotating inside a glass capillary. Originally an oversize rod was ground down to fit a selected capillary. But grinding proved impracticable. It was decided, therefore, to match one of a selection of rods against a precision-bore glass capillary, omitting the grinding operation altogether. This was successful, and the authors now keep a few dozen steel drill rods of nominal diameter 0.081 inch in stock. Whenever a new seal is required these are matched with glass capillary tubing of an inside diameter of 0.081 inch $\pm$0.0001 inch. Normally, the rod has a clearance of 0.0001-0.0002 inch in the bearing. A cup should be fitted at the top of the assembly to hold a few drops of sealing fluid. Tests have shown that the drive operated for months without wear at speeds up to 4,000 r.p.m. Di-2-ethyl hexyl phthalate was used as the lubricant.

Sommaire. Description d'un presse-étoupe utilisé dans un appareil à distiller muni d'agitateur.

Note by D. J. Trevoy & W. A. Torpey *Analyt. Chem.* **24**, Aug. 1952, 1382

A High Speed Rotary Vacuum Seal — 150/II

United States. Few seals are available which permit the introduction of rapid rotational motion into a highly evacuated system. A seal is described in this note which is satisfactory at speeds up to 18,000 r.p.m. for short periods, and has a fairly long service life at speeds up to 6,000 r.p.m. It is leaktight for pressures as low as 10^{-6} mm. Hg against atmosphere. The design provides for the use of two Klozure seals with a guard vacuum between them which is produced and maintained by a rotary pump. In this way the inner Klozure is relieved of much of the frictional wear, usually experienced, and can maintain the vacuum in the system even if the outer Klozure wears out during a run. Details of the design are given and an illustration is shown. The driving shaft is about $\frac{3}{8}$ inch diameter. All components are assembled in a cylindrical brass case which is built in two parts to facilitate rapid stripping of the seal. The outer case is attached to the evacuated vessel. Cooling

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at high speeds is advisable. For this purpose cooling water is circulated through ¼ inch copper tube soldered around the rear portion of the outer case. Klozures with rubber seats are provided in the design shown but Teflon seats may be more suitable for the outer seal, because of their high resistance to friction.

Sommaire : Joint rotatif à vide à grande vitesse ayant des performances satisfaisantes à des vitesses allant jusqu'à 18,000 t.p.m.

Note by L. R. Sitney *Rev. Sci. Instrum.* **23**, Sept. 1952, 505-506

A Flexible Vacuum Joint — 151/II

United Kingdom. Normally bellows are used where a limited relative movement is required between two large sections of a demountable vacuum system. But in the case of the ' dees ' in the Birmingham 60 inch cyclotron, which need to be moved up and down or sideways by about 1 inch this proved too costly. An alternative design has been developed, therefore, which provides for a flange on the main vacuum system, and a flange on the moving shaft, joined by a length of rubber tubing, which is supported internally by a helical wire spring. The rubber tube is clamped to the flanges by brass bands. A floating ring in the tube prevents it from collapsing. Butyl rubber with a leak rate of 0.05 μl./sec. was used in the particular case. No additional precautions were required to stop leakage. The diameter of the tube should be 10% smaller than that of the system, on which it is to go. It should be shrunk on to the flanges, if necessary swelling it first in petrol.

Sommaire : Description d'un joint spécial flexible utilisé dans le cyclotron de Birmingham.

Note by J. H. Fremlin *J. Sci. Instrum.* **29**, Aug. 1952, 267

' O '-Ring Sealed Vacuum Valves — 152/II

United Kingdom. Bellows-sealed valves are expensive, and often give trouble because brass bellows tend to crack in the presence of mercury vapour. The author describes a vacuum valve which is cheaper and simpler to manufacture than the bellows-sealed type. ' O '-rings are used to seal the seats and the glands. Two designs are shown in sketches. One is a two-way valve for use as a by-pass valve in the backing line of the diffusion pump. It is so designed that no air can be accidentally admitted to the pump during the roughing stage. The valves have been used for two years, and have proved satisfactory.

Sommaire : Un robinet à vide a été construit, muni de joints toriques pour remplacer les robinets à soufflets et éviter les fréquents ennuis dûs aux fissures des soufflets en laiton.

Report by P. A. Shatford *A.E.R.E. Rep. No.* G/M96 28.8.51

A Vacuum Cold Trap — 153/II

United States. A cold trap is described for use between the diffusion pump and the vessel to be evacuated. The cold trap is cylindrical in shape, $3\frac{3}{4}$ inch. long and $2\frac{5}{8}$ inch. in diameter, and fitted with two internal baffles. The trap is cooled by a mixture of dry ice and acetone circulating through copper tubing soldered to the outside of the wall and producing a temperature of –70°C at the baffles. The circulation of the refrigerant is due to a self-pumping effect which appears to be caused by the acetone dissolving CO_2 which evolves in the form of gas and, after having reached the necessary pressure, pushes the liquid into the reservoir to which the cooling coil is connected. The refrigerant flows in an upward direction.

Sommaire. Trappe réfrigérée de construction spéciale et montée a l'entrée d'une pompe à diffusion.

Note by J. Hino, D. K. Stevens, & H. E. Stauss *Rev. Sci. Instrum.* **23**, Aug. 1952 445-6

On the Assessment of Back-Streaming in Vacuum Systems — 154/II

See Abstract No. : 127/II

29 — MISCELLANEOUS APPARATUS — 29

Vacuum Technique — 155/II

See Abstract No. : 174/I

Production of Ammonium Nitrate by Continuous Vacuum Crystallisation — 156/II

See Abstract No. : 214/III

A Cantilever Microtome — 157/II

United States. A brief reference is made to a new cantilever microtome developed by the Battelle Institute, Columbus, Ohio. It can be used to cut sections of biological tissue as thin as 5 millionths of an inch. This is sufficiently thin to facilitate examination in the electron microscope, and it is expected therefore that the new microtome will greatly assist the study of the cell structure in plant and animal tissues.

Sommaire : La construction d'un microtome coupant des sections de tissus biologiques aussi minces que 100 Å est décrite.

Note by Anon. *Amer. Sci.* **40**, July 1952 412

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A Quick Release Seal for Vacuum Desiccators

158/II

United Kingdom. Many all-glass vacuum desiccators are sealed simply by greasing the surfaces of the lid and the body. After evacuation, and in cold weather, it is often difficult to remove a lid sealed in this manner. The use of a rubber ring is suggested stuck to one of the surfaces between lid and body. The rubber, compressed during sealing, expands slightly after release of the vacuum, and consequently the lid can be lifted away freely. Rubber of 40 Shore hardness should be used and the ring should be stuck down with a suitable rubber cement.

Note by J. H. Fremlin *J. Sci. Instrum.* **29**, July 1952, 236

Sommaire : Description d'une forme simple de joint à vide rapidement démontable pour dessicateurs à vide.

Unusual Vacuum Flasks

159/II

United Kingdom. The article describes special vacuum flasks to realise the storage of cold liquids, such as liquid oxygen to supply breathing oxygen in aircraft.

(*Bull. Int. Inst. Refrig.*)

Article by Anon. *World Refrig.* March 1952, 122-123

Sommaire : Description d'un récipient à vide spécial.

The Principles of Leak Detection

160/II

See Abstract No. : 183/I

Experiments With a Differential Pirani Gauge Leak Detector

161/II

United Kingdom. The principle of the differential leak detector is explained. With a differential Pirani gauge detector located on the backing side of a diffusion pump, where the average pressure was 15 micron Hg, the ' noise level ' of pressure fluctuations registered by the galvanometer in the detector control circuit was seven times less than that registered by a single Pirani gauge. The minimum air pressure change which could be detected by the differential arrangement was 10^{-3}mm. Hg. With the optimum leak hunting conditions employed here, this corresponds to a leak on the high vacuum side of about 10^{-8} lusecs.

(*Author*)

Report by A. H. Turnbull *A.E.R.E. Rep. No.* G/R477 March 1950

Sommaire : Construction, disposition et performances du détecteur de fuite Pirani différentiel.

Method and Apparatus for Measuring Tightness of Vessels

162/II

United States. A method of leak testing is described which is claimed to represent an improvement over the established ' pressure build-up method '. The latter method requires a vacuum pump, a valve in the exhaust line, and a vacuum gauge between the valve and the vessel to be tested. If a leak is suspected the valve is closed after pumping down and readings are taken on the gauge at regular time intervals. From these readings the amount of inleakage can be computed. The new method is appreciably faster. Normally, observations take 5-10 minutes. The vessel to be tested is placed under a hood. The exhaust line extends beyond the hood, and on the outside is fitted with an analyser which measures the quantitative concentration of any gas passing through the line. On the inside of the hood a calibrated reference leak is connected to the same line, which can be sealed off by a valve. In operation the vessel is pumped down, helium is admitted into the space under the hood and the valve connecting the calibrated leak is opened. When a predetermined concentration of the helium gas passing through the line has been reached the analyser reading (1) represents the total inleakage, *i.e.* the unknown leakage of the vessel plus the known leakage through the calibrated leak. The valve controlling the calibrated leak is then closed and another reading (2) is taken which is a measure of the unknown leakage only. The actual rate of the unknown leakage can then be determined from the following formula :—

$$\frac{\text{Reading (2)}}{\text{Reading (1)} - \text{Reading (2)}} = \text{Calibrated Leakage}$$

R. B. Jacobs & U.S. Atomic En. Comm. *U.S. Pat.* 2,608,855

Sommaire : Une méthode nouvelle rapide de recherche de fuites.

Leak Detector for Evacuated Systems and Devices

163/II

United States. An improved design of a Palladium leak detector is proposed. The known designs suffer from the following disadvantages : The permeability of palladium for hydrogen deteriorates if oxygen is absorbed on the surface. As a result the sensitivity of the gauge is reduced. Oil vapours can influence the accuracy of the readings as the hot palladium tends to dissociate these vapours and liberate hydrogen. As a result small leaks are no longer detectable. The new design is claimed to overcome these difficulties by providing for an ionisation gauge of special construction. It has a metal envelope with an anode consisting of a metal tube sealed into the wall of the envelope. The anode is fitted with a palladium cap at the inner end (within the envelope) where it faces the oxide-coated cathode. Close to the cathode is a positive ion collector. The electron discharge extending from anode to cathode heats the palladium cap to the temperature (approximately 300°C) at which it is permeable to hydrogen gas. The hydrogen penetrating the cap is ionised and attracted by the collecting electrode. This new arrangement eliminates the need for a special filament to heat the palladium cap. In its applied form, the apparatus consists of an exhaust manifold which is connected (a) to

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the pump, (b) to the vessel to be tested, and, (c) to the palladium-barrier ionisation gauge *via* a liquid air trap. Hydrogen is supplied as in other designs by placing a hood over the vessel to be tested and admitting the gas to the space between hood and vessel. Oil which may have condensed on the palladium cap, while the cold trap was out of action, can be burnt out in two minutes by opening the manifold to the atmosphere and switching on the ionisation gauge in order to heat the palladium cap. The oxides present are removed by chemical combination with hydrogen. For this purpose a small volume of hydrogen is maintained in the system, at a pressure of approximately 10^{-7}mm. Hg. As free hydrogen is continuously lost in the system by absorption and gettering effects a filament of Nichrome wire is provided in the exhaust manifold which operates at a temperature sufficiently high to decompose the hydrocarbon vapours present in the manifold thus keeping the volume of residual hydrogen constant. In order not to endanger the correct indication of the gauge by the presence of the hydrogen liberated by the filament, the temperature of the filament is controlled by a circuit which is interlocked with the gauge. As a result of this provision the palladium cap remains free from oxides and its permeability is consistent. The sensitivity of the new leak detector is given as 1×10^{-4} μl./sec.

Sommaire : La construction d'un détecteur de fuites au palladium est signalée, qui aurait une sensibilité ameliorée, par rapport aux dispositifs antérieurs.

Radio Corp. Amer. *Brit. Pat.* 663,852

Versatile Electronic Manostat

See Abstract No. : 212/III

164/II

High Altitude Research

See Abstract No. : 200/I

165/II

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30 — EVAPORATION — 30

Method for the Production of a Vacuum 172/III

See Abstract No. : 126/II

The Method of Vacuum Evaporation 173/III

United Kingdom. The vacuum evaporation method has made great progress during the last few years, in particular, where it is used for the production of thin films. Typical industrial applications of today are the aluminising of telescope mirrors, the manufacture of photoelectric cells, and the blooming of lenses. A typical application in research is the use of evaporated silver films used in Tolansky's multiple-beam interference technique. The evaporation process has to be carried out in a vacuum sufficiently low to allow for the mean free path of the evaporated atom to be large, compared with the dimensions of the chamber, in order to avoid collisions with the residual gas. A table reproduced below gives the length of the mean free path of a silver atom evaporated at various air pressures.

Pressure of Residual Air (*mm. Hg*)	*Mean Free Path of an Evaporated Silver Atom* (*cm.*)
10^{-3}	0.45
10^{-3}	4.50
10^{-4}	45.00
10^{-5}	450.00
10^{-6}	4500.00

It shows that the pressure should not be higher than 10^{-4}mm. Hg where the source-to-target distance is approximately 50 cm. Design details of evaporation chambers are given, and a workholder is described which has adjustable arms enabling the target to be fixed in almost any position. Various types of evaporation sources are mentioned, *e.g.* a graphite crucible heated by bombardment with electrons from a tungsten filament in a nearby position. In order to protect the support from sudden bursts of evaporated material, due to the evolution of gases while the charge is melting, a shutter arrangement is recommended. The higher the evaporation rate, the purer is the deposit. But too high a rate of evaporation can prove unsatisfactory. For instance, certain crystalline materials, such as magnesium chloride, give powdery deposits and poor adherence to the substrate in these conditions.

Sommaire : Revue généralisée de la technique d'évaporation sous vide d'aujourd'hui.

Article by O. S. Heavens *Lab. Practice* **1**, July 1952, 157-161

Russell Effect on Evaporated Metal Films 174/III

See Abstract No. : 215/I

The Distribution of Thin Films Condensed on Surfaces by the Vacuum Evaporation Method 175/III

See Abstract No. : 208/I

A Tracer Method for the Thickness Measurement of Thin Bi Films 176/III

See Abstract No. : 206/I

Measurement of the Thickness of Transparent Films With the Light-Profile Microscope 177/III

See Abstract No. : 207/I

Evaporation of Silver from Electro-Plated Heating Elements 178/III

United States. Normally, when evaporating silver, helical heating elements are used, formed from stranded tantalum or molybdenum wire, with small pieces of silver wire hooked-on to the filament. The authors were concerned with the deposition of silver coatings on quartz oscillator plates. Here greater precision of film thickness is required than can be obtained with the conventional method. In order to achieve this, the authors electro-plated the silver on to the heater source prior to use. Details of the process are given. Molybdenum wire of 0.030 inch diameter was wound on a mandrel $\frac{1}{4}$ inch in diameter, having a helical groove 7 inch. long, 0.010 inch deep and a pitch of 4 turns to the inch. The heater was immersed in a standard cyanide plating bath for several hours at a low current density. If necessary, the plated heaters could be preserved for a few days in distilled water.

Sommaire : L'auteur est en faveur d'une méthode d'évaporation d'argent laquelle consiste à déposer galvaniquement une quantité d'argent sur la source.

Note by L. A. Howard & C. M. Berntson *Rev. Sci. Instrum.* **23**, July 1952, 383

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The Use of a Tungsten Filament as an Evaporation Source for Silver

179/III

France. It is difficult to evaporate silver from conventional tungsten filament sources. The silver will not adhere to the filament. In order to improve this condition the authors decided to coat the tungsten electrolytically and report on a method which proved successful. As a first step the tungsten filament, wound from 0.5 mm diameter wire, was cleaned in an electrolytic bath of a sulphuric acid solution, using the tungsten as a cathode and a platinum ribbon as the anode. The current employed was 0.4 A. Subsequently, the clean tungsten wire was plated with a very thin layer of silver. In this second electrolytic process a silver wire was the anode. The composition of the bath was : cyanide of potassium (25 g.), distilled water (1 l.), and nitrate of silver (15 g.). A current of 1.5 mA was applied for 10 minutes. The silver wire was 10 cm. long. Tungsten filaments processed in this manner were used for the evaporation of U-shaped pieces of silver wire, 1 cm. long and 0.5 mm. diameter, attached to the filament. 90% of all silver globules formed during the evaporation adhered to the filament and evaporated before dropping off. Each filament could only be used once. It was not possible to use the same procedure in the case of tantalum. Silver would not adhere to it unless it was cleaned and cleaning by electrolytic means resulted in excessive brittleness.

Note by R. Roig & E. Collet *C. R. Acad. Sci., Paris* **234**, 1952, 1677-1680

O *Sommaire* : Détails d'un traitement spécial qui facilite l'utilisation de filaments de tungstène pour l'évaporation de l'argent.

Device for the Metallisation of Strips of Temperature-Sensitive Plastics in a Vacuum

180/III

Germany. The metallising of bands made from polystyrol requires special precautions in view of the heat sensitivity of the material. To avoid adverse effects it has been proposed to deposit in one operation just as much metal as can be managed before reaching the temperature limit and, should thicker deposits be required, to repeat the process as often as necessary. This drawback can be overcome, it is claimed, by the device described in this patent. It consists of a flat, cylindrical container which is evacuated and fitted with an evaporation source on the inside of the wall. The container houses a drum which can be rotated. The band to be coated is fed on to, and around its circumference, facing the container wall, by a motor-driven mechanism incorporated in the drum. In operation, the drum is rotated at fairly high speeds whereas the band is moving around the drum at a comparatively low speed. In this manner each area of the band is exposed to heating for short periods intersected by comparatively long cooling periods.

T. Baum & K. Raithel *German Pat.* 832,831

Sommaire : Dispositif à tambour tournant proposé pour la métallisation d'un ruban continu sous vide qui éviterait une élevation excessive de la température de la matière de base.

A New Device for Metallic Shadow Casting

181/III

Japan. In order to improve the precision of shadow contour in shadow casting processes a device has been developed, the main feature of which is a nozzle which guides the vapour on to the specimen at the desired angle. The device can be fixed to the baseplate of an ordinary bell jar. The evaporation source is placed out of centre. The specimen is fully enclosed on all sides by a glass cube. In the case described the distance between the evaporation source and the specimen is 5 cm. A small tube, 22 mm. long, is sealed into the cube face nearest to the evaporation source in an inclined position to give a shadowing angle equal $\tan^{-1} 1/5$. Its bore has a diameter of 0.7 mm. at the specimen end and 1.5 mm. at the source end. A shutter operated by electrical means prevents the vapour from entering the nozzle before the correct temperature has been obtained. The device has other advantages besides those for which it was originally developed : It facilitates the use of copper, molybdenum and iron, for shadowing purposes, all of which proved unsatisfactory in the past. It serves as an indicator of pressure conditions during processing. The vacuum is poor if the evaporated metal fails to pass through the nozzle. Finally, there is no longer a need for a proper point source.

Note by T. Hibi *Rev. Sci. Instrum.* **23**, July 1952, 383-385 see also *J. Appl. Phys.* **23**, 1952 957

O *Sommaire* : Description d'un dispositif spécial d'ombrage lequel donnerait des ombres plus nets et permettrait l'emploi de métaux jusqu'à présent inutilisables pour ce but.

Evaluation of Several Types of Replicas

182/III

See Abstract No. : 142/II

The Deposition of H.F. Crystal Electrodes by Vacuum Coating

183/III

United Kingdom. Normally, the electrodes used on high frequency quartz crystal plates are produced by sputtering gold onto the surface of the crystal. Due allowance has to be made for variations of the characteristic frequency in the process and specified frequencies have to be worked to. Recently interest was shown in the use of the vacuum evaporation technique for the purpose, see the work of Vigoureux and Booth, Sykes and Parrish. The author describes a specially developed evaporation plant which permits production of the electrodes complete in one process without breaking the vacuum. The chamber is exhausted by a silicon oil diffusion pump with a baffle speed of 50 l./sec. reaching 10^{-4}mm. Hg in 10 minutes time. One charge consists of 16 H.F. crystals. The crystals are placed on the circumference of a large perspex disc suitably drilled and fitted with holders positioned on a turret in the centre of the baseplate. A mask with its apertures parallel to the surface of the crystals is fitted on top of the perspex disc. To produce the initial film of approximately 0.2 μ thickness 1 g. of gold is evaporated from a source at the top of the vacuum chamber, the beam reaching the crystals normally to their surfaces. For subsequent frequency calibration a separate source is used which

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is brought near to the crystal surface, and consists of a heater holding sufficient material to process 4 crystals. Four heaters are provided to cope with the whole charge. The source is a molybdenum coil bent into a hairpin shape and emitting a beam in a solid angle of less than 2°. For measuring the frequency during operation each crystal is in electrical contact with an externally positioned test oscillator. The mechanism provided for rotating the work holder and moving the vapour source turret into position is described in detail. It is controlled by a single handle on the outside of the plant.

Sommaire : Détails d'une installation développée spécialement pour faciliter le dépôt d'électrodes sur des cristaux de quartz à haute fréquence au moyen de l'évaporation sous vide.

Article by L. Holland *Electronic Engng.* **24**, Jan. 1952, 10-13

Electron Microscope Studies of Evaporated Cadmium and Zinc 184/III

United States. The authors have developed a technique of depositing metals by evaporation within the electron microscope, which facilitates continuous observation of film formation during the operation. In a previous publication they reported on experiments carried out with silver, gold and tin. None of these three metals showed the phenomenon of ' critical density '. This term describes a condition applicable to some metals, which calls for a well-defined minimum density of the incident beam, before a permanent deposit is obtained. Its value depends on the temperature of the substrate. Below that value all atoms hitting the substrate are assumed to be reflected or re-evaporated. The present experiments were carried out with cadmium and zinc on substrates at room temperature. These metals were known to exhibit the effect of critical density and therefore expected to show a structure of the initial film different from that of gold, silver and tin. The results of the experiments are shown in a series of electron micrographs. Formvar films were used as substrate. In the early stages the particles had a regular shape, but as they continued to grow, their shapes became irregular. In the final stages the particles linked up with each other and formed irregular chains. Normally, few new particles would appear subsequent to the initial stage. But, if at any time the deposit had been exposed to air, new nuclei would form readily. This may have been due to the formation of an oxide on the particles deposited prior to exposure which inhibited further growth. The examination of the build-up of zinc deposits in the perpendicular direction was facilitated by shadow casting with chromium. Examining the shadow-cast specimens a number of particles were traced, shaped like flat platelets, which had been transparent to the electron beam. Also, a number of triangular particles could be seen which appeared to stand on one edge, at an angle of about 60° with the substrate. A significant feature of the present experiments was the sudden appearance of the initial particles on the substrate, some of them as large as 200 Å when first noticed. This phenomenon appears to be a characteristic of critical density, *i.e.*, a certain minimum density of the beam is required to form the first nuclei. Once they have been formed, they grow with great rapidity.

Sommaire : Recherches sur la physique de la formation de films à l'aide du microscope électronique.

Article by R. S. Sennett, T. A. McLauchlan & G. D. Scott *Canad. J. Phys.* **30**, Sept. 1952, 370-372

Electron Diffraction in Some Cubic Crystals 185/III

See Abstract No. : 209/I

Some Miscellaneous Vacuum Coater Techniques 186/III

United States. A variety of auxiliary techniques are described which were developed in connection with the operation of a vacuum evaporation plant of standard commercial design. The bell jar was 18 inch. diameter and 28 inch. high. The diffusion pump was fitted with a water-cooled baffle and a valve which permitted breaking the vacuum without stopping the pump. Rough pumping took about 90 seconds to reach a pressure of 100 μHg. The operational pressure was about 2×10^{-5} mm. Hg. Two separate Variac-controlled low voltage circuits were provided, facilitating the evaporation of two substances simultaneously or in succession without breaking the vacuum. A special source was developed for the evaporation of fluorides. The boat was made from tantalum or platinum sheet, 0.005 inch thick, and formed by rolling the sheet around a rod of $\frac{1}{4}$ inch diameter, slotting it in the centre to take about 2-3 g. of the material to be processed. The terminals were of pure copper fixed to the busbars by stainless steel screws. Platinum appeared to be more satisfactory than tantalum in this application. For the evaporation of aluminium the source design was altered to carry a small boat of re-crystallised alundum, sprung into the tantalum trough. Aluminium, silver, copper, nickel and germanium were successfully evaporated from this source, but iron appeared to attack it. The cleaning of the bell jar after use is difficult, in particular, when aluminium has been evaporated. The latter adheres firmly to the wall of the jar. It is recommended that 0.5 g. of fused potassium chloride is evaporated before operations begin. The salt forms a thin film on the glass which serves as a water-soluble substrate for the aluminium, and by wiping the wall with a damp cloth immediately after operations the aluminium can be easily removed. Finally, experiments are mentioned which aimed at closing the pores of evaporated fluoride coatings by depositing a very thin layer of fluorothene-W wax on top, but no conclusive results had been obtained by the time the report was closed.

Sommaire : Conseils pratiques concernant un nombre de problèmes en rapport avec l'opération d'un appareil à évaporation sous vide, standard, pour le traitement de lentilles.

Report by A. E. Cameron *A.E.C. Rep. No.* K-909 12.5.52

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Evaporated Magnesium Fluoride Films

187/III

Japan. A study was made of the factors which influence the structure of anti-reflection films produced by the evaporation of magnesium fluoride. The magnesium fluoride was evaporated at about 10^{-7}mm. Hg from a basket-type evaporation source and deposited on a small piece of crown glass, placed 8 cm. away. Half of the substrate was masked during the process. After deposition the transmissivity of the coated and uncoated parts of the substrate were measured with monochromatic light at normal incidence with the help of a selenium cell. The optical thickness of the films was approximately $\frac{1}{4}\lambda$ for maximum transmissivity. The ratio of the transmissivity of the coated glass to that of the uncoated glass was determined by a formula, given in the article, to have a maximum value of 1.029 at 6,200 Å. The refractive index was calculated to be 1.38. If the substrate was kept at room temperature during deposition, the refractive index was found to be the same as that of bulk magnesium fluoride, *i.e.* 1.39. But, if the substrate was at a higher temperature, the results were erratic. In that case the maximum value for the ratio of transmissivities was oscillating between 1.033 and 1.031 and the refractive index was 1.37. This seems to indicate that deposits on glass at room temperature are free from pores, whereas films deposited on glass at elevated temperatures are porous due to the formation of a crystalline structure.

Article by N. Morita *J. Sci. Res. Inst. Japan* **46**, Sept. 1952 127-129

Sommaire : Recherche sur les facteurs de traitement influençant la structure et la qualité de couches antiréfléchissantes produites par évaporation de fluorure de magnésium.

The Application of Aluminium Reflecting Films to Glass and Plastics by Thermal Evaporation

188/III

Canada. A study is presented of the fundamental principles which govern the deposition of aluminium films upon various materials. The article is divided into two parts. Part I deals with films on glass supports and Part II (to be published in the next issue) with films on plastics. The conclusions and notations are extended wherever possible to the commercial aspect of evaporation. (*Author*)

Article by S. Bateson *Vacuum* **2**, Oct. 1952 365-376

Sommaire : Etude des principes gérant le dépôt d'aluminium sur verre ou plastiques par la méthode d'évaporation avec référence particulière à la production à l'échelle industrielle.

The Protective Properties of the Natural Oxide Films on Aluminium

189/III

See Abstract No. : 223/I

Preparation, Properties and Optical Applications of Thin Films of Titanium Dioxide

190/III

United States. The paper presents data on the optical properties, structure and oxidation of evaporated titanium films and discusses the structure, properties and applications of titanium dioxide coatings prepared by heat oxidation of vacuum deposited titanium and by decomposition of titanium tetrachloride with atmospheric water vapour. It is shown that the rate of deposition and the pressure in the evaporation unit influence greatly the structure and optical properties of evaporated titanium films, and of titanium dioxide coatings prepared by heat oxidation of evaporated titanium. Pure titanium films can only be deposited by fast evaporation at good vacuum. Slow evaporation at poor vacuum results in TiO-TiN films with cubic structure. The oxidation of pure titanium films in air at 400° to 450°C results in titanium dioxide films with rutile structure and high n-values while ' poorly deposited titanium films ' oxidise at the same temperature to titanium dioxide films with anatase structure and much lower n-values. Rutile is especially suitable as a top film in multilayer combinations. Titanium dioxide films produced by fuming titanium tetrachloride on to hot glass are amorphous if the glass temperature is kept below 280°C and consist of anatase at higher temperatures. An anodic method for applying reflectance-increasing film pairs of aluminium oxide and titanium dioxide and aluminium mirrors is described. (*Author*)

Article by G. Hass *Vacuum* **2**, Oct. 1952 331-346

Sommaire : Discussion des propriétés optiques, structure et oxydation de titanium evaporé.

The Production and Electrical Properties of Thin Metal Films

191/III

France. The article gives a description of various methods employed to prepare films, the properties of which have been subsequently investigated from the following angles : The relation between thickness and electrical conductivity of the films, the effect of the nature of the substrate and the nature of the metal on film structure and growth, and the influence of temperature. The metals used in the experiments were gold and copper, the substrates essentially silica, plexiglass and monocrystals of NaCl, KCl, Kbr and NO_3Na.

Article by G. Ranc *Le Vide* 7, July-Sept. 1952 1211-1219

Sommaire : Les facteurs influençants la conductivité électrique de couches minces métalliques ont été étudiés.

Filters for the Infra-Red Region

192/III

United States. The authors report on the production and performance of infra-red transmitting films deposited by the vacuum evaporation method and prepared from tellurium, bismuth, antimony and magnesium oxide. Filters of this kind are used for the removal of stray radiation and the segregation of narrow bands in radiometric measurements. The films should be opaque in the visible region and should have short wavelength cut-offs in the infra-red. The antimony films were deposited on a lithium fluoride plate and the bismuth films on a calcium fluoride plate. The former were opaque at 1 μ and gave a transmission of 75% at 3 μ. The

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latter cut off at 2.2 μ, if of sufficient thickness, and gave a transmission varying from 60-70% at 5 μ. This variation in transmission appeared to depend primarily on pressure conditions, *i.e.* on the presence of oxygen during deposition. Harris and McGinniss have shown previously that gold black can be made highly absorbing up to 30 or 40 μ, if no oxygen is present in the evaporating chamber, but can be made a very good filter with the cut-off near 1 μ, if oxygen is present. The most rapid increase in transmission from the cut-off point could be obtained with tellurium films on rock salt. These films were deposited at a pressure of 10^{-3}mm. Hg. Pfund, reporting on tellurium films in an earlier publication, used a different method, and this explains why the films described here show different qualities, *i.e.* a sharp rise in transmission and high resistance against abrasion. By combining cellophane filters and tellurium films on glass one can produce a filter which is transparent in the range from 1.4 μ to 2.8 μ. The magnesium filters described in the article were produced by an atmospheric process.

Article by E. K. Plyler & J. J. Ball *J. Opt. Soc. Amer.* **42**, April 1952 266-268

Sommaire : La production et les caractéristiques de films transparents à l'infra-rouge, produits par évaporation sous vide, utilisant du tellure, du bismuth, de l'antimoine et l'oxyde de magnésium, sont discutées.

31 — CATHODIC SPUTTERING — 31

Deposition of Conducting Films on Glass

193/III

United Kingdom. This article describes the same work as that reported in Abstracts 151/III and 152/III, but gives more details. The American process for the production of electrically conducting films consists of heating a sheet of glass almost to softening point, and then spraying it with a solution containing fluoride of tin. The film left on the glass is probably plain tin oxide, its light transmission is about 80%, and its electrical resistance 100 ohms between the opposite sides of a square. The first conducting films prepared in the National Physical Laboratories were of a quality similar to that of the American product, but the processing was different, *i.e.* the films were deposited in vacuum by cathodic sputtering. When the Ministry of Supply asked for a product with a lower electrical resistance, the British investigators studied the possibilities of pure metal films as distinct from metal oxide films. It was found that the resistance value of a multilayer film, made up from a bottom layer of certain oxides such as lead or bismuth oxide and a top layer of gold, could be reduced, after suitable heat treatment, to approximately 10 ohms per square. The transmission of these films for white light is just under 75%. This is less than that of the American film, but the British film may be directly operated from a 12 V. battery, instead of from a separate high voltage generator required for the American film. The thickness of the individual layers is approximately $\frac{1}{4}$ of a millionth of an inch. The amount of gold required for coating a sq. ft. of glass does not exceed the value of 1d. The article concludes with a short survey of the potential uses of conducting films, for instance, in vacuum apparatus, such as fluorescent lamps or mercury glass rectifiers, where it would be an advantage to have conductive areas on certain parts of the inner glass surface.

Article by Anon. *Chem. Age* 5.7.52 13-15

Sommaire : Détails de travaux expérimentaux en U.S.A. et en Angleterre concernant le développement de films transparents et conductibles.

Transparent Conducting Films

194/III

United Kingdom. Brief remarks are made on the behaviour of gold when used for the production of thin electrically conducting films. The films were deposited by cathodic sputtering and the deposits were approximately 60 Å in all cases. It was found that deposition on glass resulted in a resistivity and optical absorption higher than that calculated from the respective data of the bulk metal. The transmitted colour was blue-green. On heating the colour changed first to blue and then to pink. The method used for cleaning the glass had a marked effect. Cleaning with dry precipitated chalk gave low resistance, low absorption and a straw colour. Chemical cleaning gave high resistance, high absorption and a blue colour. Providing zinc oxide as a substrate gave a pale straw colour, low resistance and small absorption. If the latter (compound) film was heated for a few minutes at about 200°C its resistance and absorption could be reduced still further to values approaching those applicable to the bulk material. In the following table, the transmission factor (T), the absorption factor (α) (both for white light), and the resistance (R) between two opposite edges of a square of film are given for two different supports as shown :—

Substrate	*R*	α	*T*
Chalk-Cleaned Glass	60–70	24	62
100 Å Bismuth Oxide, on Glass, after Heat Treatment	10	13	75

Letter by E. J. Gillham & J. S. Preston *Proc. Phys. Soc.* **65B**, Aug. 1952 649

Sommaire : Brève note sur certains phénomènes observés en développant des films, transparents, conducteurs d'or.

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33 — IMPREGNATION — 33

Vacuum Impregnation of Castings 195/III

United States. Porosity in castings can be due to poor design, defects from physical or chemical causes, or improper foundry practice. Impregnating the castings will appreciably reduce the number of rejects for porosity. A good impregnant should be of low viscosity, low volatility, and should dry with little or no loss of volume. It should allow for heat treatment of the component up to 300°F and should have no effect on machineability. Satisfactory materials are resin and resin-oil combinations, such as styrene with linseed oil or dehydrated castor oil. Five methods of impregnation are mentioned : (1) Immersing the casting in the impregnant which is kept in a vessel to which pressure is applied. (2) Internal-pressure method, by which the components are sealed off except for one port. (3) External pressure method. (4) Vacuum impregnation by batch immersion followed by pressure, and (5) the use of vacuum only. Of all these, No. (4) is the preferred method at present. The parts are cleaned and degreased and then heated to 250°F for about 2 hours. After cooling down they are loaded into the vacuum chamber and a vacuum is drawn to 29 inch. or more. This is maintained for 20 minutes. The capillary ducts of the casting should now be free from air and moisture and should offer easy access to the impregnant. The impregnant is fed into the tank until all components are covered while the vacuum pump continues to draw. Then the vacuum pump is stopped and pressure applied in the tank (approximately 60-100 p.s.i.) for a period of 20-40 minutes. Finally, the impregnant is withdrawn and the castings are removed, drained and rinsed in an acetone solution. Baking or air curing follows. Magnesium castings require a somewhat different treatment.

Article by L. W Hull *J. Metals* Jan 1952 30-32

Sommaire : Les méthodes connues d'imprégnation de pièces de fonderie métallique sont étudiées et les mérites comparatifs de l'imprégnation sous vide sont indiqués.

Impregnation of Masts 196/III

Denmark. A description of the two methods of impregnating fir with tar oil, after removal of bark, and drying in open air : In a boiler containing the oil under pressure, and in a boiler under low pressure and filled with oil after the air in the timber has been (partially) removed. In the case of spruce, the bark is not removed, as impregnation must be carried out along the axis of the trunk. The usual liquid is copper sulphate, but recently NAF salt, a mixture of sodium, arsenic and fluorine compounds, has been employed. Examination of masts in the field is discussed and some photographs of fungus attacks are shown.

(*Science Abstracts*)

Article by V. Hallund *Elektroteknikeren* **48**, 22.5.52 172-6

Sommaire : Méthode d'imprégnation de bois sous vide.

34 — FUMIGATION — 34

Study of Vacuum Fumigation Techniques 197/III

United Kingdom. A short note in the Annual Report of the Pest Infestation Research Board reports on tests comparing the quality of various low pressure fumigation methods with treatment at atmospheric pressure. A chamber of about 1,700 litres capacity was used situated in an air conditioned room kept at 15°C and described in detail elsewhere in the report. The fumigant was methyl bromide and the products tested were bagged wheat and boxed dates. Preliminary results seemed to indicate that vacuum techniques offer no advantages in the case of granular products such as wheat, but in the case of dates penetration in vacuum was speedier than at atmospheric pressure. Introducing only the fumigant after evacuation and keeping the chamber at constant low pressure for a period, resulted in better penetration than the method of introducing a mixture of the fumigant and air into the evacuated chamber and, at the same time, bringing it up gradually to atmospheric pressure. The tests with bagged wheat will be continued.

Ann. Rep. by *Pest Infestation Res. Board D S. I.R* 1951 28

Sommaire : Brève note sur la fumigation de matières alimentaires sous vide.

36 — DRYING — 36

Design Features of Modern Vacuum Drying Plant 198/III

See Abstract No. : 199/I

Frozen Concentrated Pineapple Juice 199/III

See Abstract No. : 216/III

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Falling Film Evaporator

200/III

United States. A multi-stage evaporator is described which has been specially designed for operation at low pressures and temperatures. It is different from the so-called multiple effect evaporator where the hot vapours of one stage are used to heat the processed material in the next. In the present design all stages have a common heat supply and vapour exhaust and units of identical construction are used for each stage. Each unit consists of a vertical tube chest, the tubes communicating between an upper and a lower chamber. The liquid to be processed is fed into the upper chamber and is directed from there through short removable tubes to the inner walls of each evaporator tube in order to form a falling film. At the bottom end of the tube chest the unvaporised liquor falls into a sump, whence it is pumped through a riser pipe to the top chamber for re-circulation through the evaporator. The vapours are withdrawn through a drum fitted to the bottom chamber, which imparts a centrifugal motion to the vapours and causes entrained droplets to sweep along the walls of the drum, until they fall into a sump in the drum. The concentrate collecting in this sump is pumped into the top chamber of the second stage unit. The rate of evaporation in the first stage is higher than in all subsequent stages. Thus the second unit deals with a liquid of considerably higher density than that of the fresh liquid introduced into the first unit. Variable speed pumps, valves or other devices may be employed for maintaining uniform or providing variable operating conditions. Two embodiments of the invention are shown.

J. A. Cross & Majonnier Bros. Co. Inc. *U.S. Pat.* 2,570,211

Sommaire : Détails d'un évaporateur à étages multiples de construction différente de l'évaporateur communément connu pour la concentration de liquide aux températures comparativement basses sous vide.

A Laboratory-Scale Continuous Vacuum Flash Evaporator

201/III

United States. The evaporator described here was specially developed for the removal of volatile flavour constituents from fruit *purées* and juices and consists essentially of a metering pump, a steam jacketed stainless steel tube, ¼ inch wide and 9 ft. long, serving as a preheater, a steam heated evaporating tube, a cyclone vapour-liquid separator, a spiral tube surface condenser, an after-cooler and two receptacles, one for the residue and one for the distillate. Dry ice and liquid air traps were fitted to the vacuum line. After pumping down the metering pump was set for the desired processing rate (up to 1 litre per minute), steam was introduced into the jacket of the preheater (1 atmosphere) and into the jacket of the evaporator tube and the temperature of the vapour emerging from the evaporator tube was checked on a thermometer. At a temperature of 35-40°C corresponding to 42-55 mm. Hg water containing the volatile flavours was removed in a single-pass operation at a rate of 5 to 50% (based on weight of feed material) in the case of thick *purée* and 5 to 80% in the case of juice. A table, reproduced below, gives the essential performance data of the apparatus.

Feed Rate and Percent Water Evaporated by Continuous Vacuum Flash Evaporator

Material Processed	*Feed Rate ml./min.*	*Water Evaporated %**
Water	100	75
Water	200	37.5
Water	400	21.0
Water	1,000	15.0
Tomato Juice	160	55.0
Tomato Juice	415	25.1
Strawberry Puree	125	38.0

* Based on Weight of Feed Material.

The amount of volatile organic material which could be recovered by flash vaporisation was assessed experimentally by distilling a solution containing 0.005% ethanol and another containing 0.005% phenol. These compounds were chosen because they differ greatly from water in their behaviour during distillation. This concentration was chosen as it corresponds approximately to that of the volatile organic flavours in fruits. In the case of the ethanol solution it was found that an evaporation of 10% of the feed removed approximately 55% of the alcohol. In the case of the phenol solution 10% of phenol was recovered when 10% of the feed was evaporated. In another series of experiments *purée* samples obtained from several varieties of strawberries were stripped by evaporating approximately 40% of water (based on the original weight of the *puree*). The residue was diluted with distilled water to the original volume and stripped again. Each distillate was then concentrated in a Stedman fractionating column until a final distillate was obtained equal to 1/30 of the weight of the original *purée*. These concentrates were then analysed for carbon representing the essence. After analysing the distillate from a third stripping it became clear that nearly all volatile organic substances which could be recovered from this material had been obtained in the first two strippings and that over 80% of the total was recovered in the first stripping alone.

Article by K. P. Dimick & B. Makower *Food Technol.* 5, 1951, 469-517

Sommaire : Un évaporateur à ' flash ' continu, de taille de laboratoire est décrit et sa performance determinée concernant l'élimination de composés aromatiques etc.

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37 — METALLURGICAL PROCESSES — 37

Use of the Hollow Cathode and the Vacuum Furnace in Spectroanalysis 202/III

See Abstract No. : 216/I

An Automatic Surface Follower for Measuring the Rate of Evaporation of Molten Metals Undergoing Vacuum Distillation 203/III

United States. In the past the measurement of rates of evaporation of metal at reduced pressures and relatively high temperatures was difficult. It was not possible to measure the rate at any particular moment; only averages could be determined. No means were available for measuring the temperatures immediately above and below the evaporating surface. Both these zones are important as the rate at which heat for vaporisation is transferred to the surface is determined by the temperature gradients in the metal near the surface and in the vapours immediately above it. The authors developed a device which automatically locates the surface of molten metal in an evacuated vessel and follows the surface level as it falls during evaporation. As long as the vessel is of uniform shape from top to bottom, plottings of the position of the evaporation surface against time give a direct indication of evaporation rates. The device is mounted on a baseplate which is fitted to the top of the still. The base plate carries the main carriage of the instrument by means of a large bellows. The main carriage supports a second carriage by means of a smaller bellows. A thermocouple tube is lowered through the latter into the furnace. The main carriage carries the control electrode, which consists of a quartz tube fitting closely over a carbon rod that projects slightly out of the tube in order to make contact with the surface of the liquid metal. The carbon rod is connected electrically to a slide-wire potentiometer mounted on the supporting column of the main carriage. If surface temperature only is to be measured thermocouple and electrode tip are set exactly level. The follower unit is operated by an electric motor. When the electrode contacts the metal surface a magnetic clutch stops the motor. As soon as contact is broken, the motor starts up again. The overall travel of the main carriage is 3½ inch. Details of the electrical control circuit are given. The maximum speed of the follower electrode is 1.06 cm./minute. In the case considered this corresponds to an evaporation rate of 345 g./min. The performance of the follower unit is shown in tables, one of which (reproduced below in abbreviated form) records the conditions encountered when evaporating zinc.

*Performance of Surface Follower Unit**

Time, min.	*Level mv*	*Diff. mv*	*Wt for Interval, g.*	*Total wt of Zn Evaporated, g.*
0	40.9	0.0	—	—
5	40.4	0.5	30.783	30 783
25	35.0	1.3	80.035	363.237
50	24.6	2.2	135.443	1003.514
75	11.3	2.2	135.443	1822.330

* Total surface displacement 40.9-9.6 = 31.3 mv
Total weight of Zn evaporated 7066¼5139 = 1927 g.
Calibration : 1927 g/31.3 mv = 61.565 g./mv
Rate of evaporation at 4 kw = 17.24 g./min
Rate of evaporation at 5 kw = 31.33 g./min

In the same experiment it was found that the temperature is 34° cooler at a point 2.4 cm. below the evaporating surface.

Sommaire : Description d'un instrument spécialement développé pour mesurer la vitesse d'évaporation de métaux en fusion en cours de distillation sous vide.

Article by M. J. Spendlove & H. W. St. Clair *Rev Sci. Instrum.* **23**, Sept. 1952 471-475

A High Temperature Sintering Furnace 204/III

United Kingdom. Normally bars of refractory materials such as molybdenum, tungsten or tantalum are sintered by passing electric current through the processed material itself. This method of heating is unsatisfactory because the ends of the bar, where connection is made with the leads, remain cooler than the centre section. Thus external heating is preferable and the author reports on the construction of a furnace which facilitates this. The furnace takes specimens up to 100 g., 75 mm. long and 15 mm. diameter. It is designed for an operational temperature of up to 2,400°C, and processing in a hydrogen atmosphere or in vacuum. The absence of all refractories as constructional materials greatly assists operation in vacuum. The heater consists of a fabricated tube 325 mm. long and 33 mm. wide. Its central section is made from tungsten sheet, 0.6 mm. thick, and the end portions from molybdenum sheet, 0.5 mm. thick. In order to prevent distortion at high temperature the tube is stiffened with two spirals of 1 mm. tungsten wire, one inside and one outside the tube. The upper end of the heater tube is bolted to a water-cooled top plate which is carried by two copper tubes which facilitate electrical connection and serve as the water in- and outlet. The bottom of the heater tube is connected to a copper pipe by a flexible lead. The specimen is suspended from a molybdenum wire and is free to centre itself by gravity inside the heater tube. It can be raised and lowered by means controlled

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from outside. Radiation shields are provided. The furnace chamber consists of a copper cylinder 3 mm. thick, 50 mm. long and 150 mm. diameter. It rests on a brass plate carrying an inlet for hydrogen, and is fitted to the top plate by a brass flange carrying an ' O '-ring. The pumping unit—a 50 mm. diffusion pump and a mechanical fore pump—is connected to the top plate. The furnace chamber is cooled by a water spray. The water is collected in a gulley surrounding the furnace chamber, and filled with sand in order to control the amount of water drained off and collecting in a receptacle which cools the bottom of the furnace. 700 A (11kW) is required for operation in hydrogen and 550 A (7kW) for operation *in vacuo*. The temperature is measured with an optical pyrometer and the pressure with an ionisation gauge. The operational pressure is 5×10^{-5} mm. Hg. Two sintering processes can be carried out in a day, operating in hydrogen, and one, operating in vacuum. Cooling from 2,300°C requires less than 2 minutes. The difference in temperature between the centre and the ends of the 75 mm. long hot zone is about 50°C in 2,000°C. The life of the heater exceeds 50 runs, *i.e.* over 80 hours at 2,300°C.

Article by J H. Rendall *J. Sci. Instrum.* **29**, Aug. 1952 248-250

Sommaire : Un four à fritter pour des températures dépassant 1,700°C qui peut être chauffé et refroidi en un temps extrèmement court en raison de l'absence de matières réfractaires.

38 — DISTILLATION — 38

On Molecular Distillation of Fats and Oils

205/III

Germany. Due to the evolution of efficient pumps and measuring instruments, molecular distillation has made great strides in the recent past. Normally the operational pressure in molecular distillation processes is approximately 10^{-3} to 10^{-4} mm. Hg. No boiling occurs in molecular distillation, evaporation takes place from the surface of the distilland only. In order to offset the low thermal efficiency of the molecular still the condenser surface is usually only some 100°C cooler than the evaporating surface. The distance from the evaporator to the condenser is governed by the length of the mean free path of the evaporated molecules, which is inversely proportional to the prevailing pressure and the molecular weight. It is usually between 35 and 40 mm. The ordinary glycerides have a mean free path of several centimetres in the range of 10^{-3} to 10^{-4} mm. Hg. Molecular distillation must be carried out at temperatures which prevent decomposition of the distilland because even slight decomposition will produce flashing and cause appreciable fluctuations of the pressure. Another factor to consider is turbulence. Various evaporator designs are described including the falling-film and Hickman's rotating frustro-conical evaporator. Heating is usually effected by electrical means. A discussion of vacuum pumps and pressure reading instruments suitable for use with the molecular still follows. Methods of degassing processed materials are mentioned. In the second section of the article details are given of various stills specially developed to serve (*a*) laboratory purposes and (*b*) industrial production. Molecular distillation of fatty acids and their esters is by now a properly established procedure in industry. An outline is given of the molecular distillation of palm oil containing a large amount of carotin. At 170°C the fatty acids are recovered, in the second fraction the carotin and in the third and fourth the glycerides. The residue consists of unsaturated components of high molecular weight. A similar account is given of the processing of coconut fat.

Article by K. H. Martens *Seifen, Oele Fette Wachse* 77, 10.10.51 491-494 & 77, 24.20.51, 517-519

Sommaire : Revue générale des lois de base de la distillation moléculaire, des constructions typiques existantes et des résultats typiques pour une ou deux huiles végétales, telles qu'huile de palme ou de coco.

Studies in High Vacuum Evaporation. The Falling Stream Tensimeter

206/III

United States. In a closed still molecules evaporate and re-condense at a rate which reaches equilibrium. In an open-path high vacuum still, and its extreme form the molecular still, there is completely non-equilibrant distillation, termed projective distillation, *i.e.* a state of ' net ' evaporation exists representing the excess of outgo over return. It has been found that liquids often refuse to evaporate at rates and temperatures expected of them in the circumstances. This has been traced back to the existence of a repressive skin forming on the surface of phlegmatic liquids. Thus, operating with vacuum stills, it is difficult to assign fixed relative volatilities to binary test mixtures and equally difficult to decide what is the separatory power of one unit of projective distillation. There exists no definition of a theoretical molecular plate. Knudsen reported that clean mercury showed an evaporation coefficient near unity, dirty mercury a much lower evaporation coefficient. It was the object of the studies reported in these articles (see also Abstract Nos. 207/III, 208/III, 209/III) to investigate whether other materials behave similarly. The first task was to obtain a clean surface of the processed material. This was achieved with an instrument described in the present article, called the falling-stream tensimeter. It consists essentially of a jet surrounded by a tubular condenser, and is thus a falling film molecular still where the film is backed by a column of liquid, instead of a heated metal surface. The condenser is kept at a temperature 100°C below that of the stream and is divided into two condensing zones by alembics. Vapour disengaging itself from the central stream of the liquid proceeds radially outwards until it reaches the condenser wall. The condensate is collected by the two alembics and withdrawn for measurement or analysis. As long as the temperature of each zone and the rate of distillation per unit area of stream are substantially the same, it can be assumed that the temperature and composition of the surface of the distilland are substantially the same as the interior. Working drawings of the tensimeter are shown. The

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test mixture used was 30 Mole % di-2-ethyl-hexyl phthalate in di-2-ethyl-hexyl sebacate. A typical series of measurements at various temperatures is shown in Table IV, partly reproduced below.

Rate of Distillation and Relative Volatility of a Liquid Containing 29.4 Mole% EPH in EHS

Temp.	*Upper Condensing Zone*			*Lower Condensing Zone*			n_D^{25} *of Condensate*			
t	A	Θ	r_u	A	Θ	r_l	Upper	Lower	y	α
100.7	9.50	2790	0.357	14.57	3615	0.359	1.4720	1.4721	0.696	5.50
120.9	9.59	496	1.98	14.63	630	2.03	1.4702	1.4702	0.646	4.40
140.7	9.59	117	8.37	14.63	146	8.73	1.4682	1.4681	0.589	3.44
161.5	9.59	31.9	30.6	14.63	39.9	31.8	1.4661	1.4660	0.530	2.71

These experiments yielded two important results : (1) The two liquids examined evaporated with a coefficient of unity from clean new surfaces, and (2) the relative volatility increased with a reduction of temperature. Assuming that these results are generally applicable, it can be concluded that tests on molecular stills with binary mixtures to determine plate efficiency are only reliable if the exact temperature and relative rate of evaporation are taken into account. Further, the measures, normally taken to keep the fluid in a diffusion pump clean, are necessitated by the abnormally high relative volatilities exhibited by volatile contaminants under very slow conditions of evaporation at the intake of the pump.

Sommaire : Détails d'un distillateur spécial appelé ' tensimètre à courant tombant ' qui facilite la production et l'évaluation de la qualité d'une surface de matière évaporée sous vide.

Article by K. C. D. Hickman & D. J. Trevoy *Industr. Engng. Chem.*, **44**, Aug. 1952 1882-1888

Studies in High Vacuum Evaporation. Projective and Equilibrium Vapour Liquid Relationships for Two Binary Systems over an Extended Range

207/III

United States. This article reports on investigations extending the results of the experiments reported above (Abstr. No. 206/III). Measurements were taken over the entire composition range of the di-2-ethyl-hexyl-phthalate di-2-ethyl-hexyl-sebacate mixture. In addition, another binary mixture di-n-octyl-phthalate di-2-ethyl-hexyl-sebacate was investigated which has a lower relative volatility than the former. The latter mixture is frequently used as a test fluid for molecular stills. All experiments were carried out at a pressure of less than 1 μ Hg. The same falling-stream tensimeter was used as employed in the previous investigation, but a large mercury thermo-regulator was inserted in the feeding line to control the heat input. Binary solutions ranging from 10-90 mole % in 10% steps were distilled in the range of saturation pressures 1-100 μ Hg. Results are given in tabular form. It was established that the system EHP-EHS, is suitable for the evaluation of single-stage molecular still performance since the relative volatility changes only with temperature. It was shown in the previous article that the rate of distillation deviates at higher pressures from that predicted by the Knudsen equation. As this may be due to self-obstruction of the vapours, extensive experiments were undertaken, varying both stream diameter and distance from stream to condenser. The results showed that these variations did not alter the measured rate of distillation or the composition of distillate. In all these experiments it was assumed that the condensation coefficient was unity. Distillation in the falling-stream tensimeter is of the projective type. In order to check whether the composition of the projective distillate from a solution is identical with that obtained by equilibrium distillation, experiments were carried out using an equilibrium still of a design fully described in another article (see Abstract No. 209/III). The operational pressure was less than 10^{-4} mm. Hg. It was found that the values of relative volatilities were largely identical below 5 μ Hg saturation pressure, but differed markedly at higher rates and pressures, the equilibrium still producing the higher values. Investigations of the second test mixture, NOP-EHS, by projective distillation in the temperature range from 100°-180°C showed that changes in the composition resulted in considerable changes in the relative volatility at the same temperature. Thus, no mean value of the relative volatility independent of composition exists for this material in the range of temperatures studied and the mixture cannot be considered suitable for testing vacuum stills.

Sommaire : Continuant les travaux décrit dans le résumé précédant des mesures de performances de deux mélanges binaires, d'une composition rélative variée, ont été faites dans un tensimètre à courant tombant et comparées avec les conditions dans un distillateur à équilibre.

Article by D. J. Trevoy *Industr. Engng. Chem.* **44**, Aug. 1952 1888-1892

Studies in High Vacuum Evaporation. Surface Behaviour in the Pot Still

208/III

United States. After having examined the properties and behaviour of clean new surfaces of certain liquids (see Abstract Nos. 206/III and 207/III), the author studies the properties of the ' ordinary ' surface of an evaporating liquid. If a solid with a clean surface is melted, contaminants will at once come to the surface. The dross forming on the surface has to be removed continuously by scraping or overflowing. Liquids as a class therefore can be considered to be self-soiling. A typical example for distillation without any provision of overflowing is the pot still. It forms a ' sink ' from which nothing non-volatile escapes. Its evaporating surface can be assumed to be contaminated and consequently, this type of still can be expected to have a low coefficient of evaporation. Another type of surface which has to be considered is the ' depleted ' surface, *i.e.* a surface which has lost its more volatile constituents. An absolutely clean surface cannot be depleted. Thus one can define the ' ordinary ' surface in vacuum distillation as a mature and contaminated surface which is also depleted. From these considerations two surface behaviours can be deducted. One is called ' torpid ' and evolves from a mature surface that becomes contaminated and then depleted. It is conspicuous for the absence of convective movements and its rate of evaporation is greatly depressed. The other, the ' pristine ' surface,

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is rich in hotter and more volatile molecules. This surface is in rapid motion. Where both surfaces are present the author speaks of a ' schizoid ' surface. To examine these conditions more thoroughly the author developed special equipment and conducted a number of experiments on the following lines : One litre of 2-ethyl-hexyl-phthalate was placed in a 2-litre flask and vacuum applied. Heating the liquid, the surface became turbulent after a short-period. 24 hours later the turbulence was still pronounced, but there was also a large area with no motion in it at all. After about a week the whole of the surface was entirely calm. Where drops of distilland fell on to the surface a crater was formed which was turbulent and met the calm surface with a steep barrier. From this crater vapour escaped, cooling the liquid. The experiments were repeated with other materials, and the same effects were produced at varying degrees. In a further experiment a combination of flasks was used with connections between them, which, due to the level difference between torpid and working area, and by means of a stirring action, allowed for the torpid area to be overflowed from one flask to another. In this manner a clean working surface could be obtained, in a period of 60-90 seconds. These observations led to a classification of surfaces and their behaviour as shown in a table, reproduced below :

Criteria for Surface Identification

Test	*Working*	*Torpid*
Appearance	Rapid Motion	Still or slow motion
Immersion heater (torpidity tester)	Increases Motion	Causes crater or explosion of vapour
Ascending hot currents	Merge with and disturb surface	Rejected by surface
Ventilation	Self-automatic	By ' teardrop ', giving ' pincushion ' cooling

By means of an anemometer probe the author succeeded in establishing the increase in temperature required to free vapour from the torpid area. The results are recorded in tabular form covering three types of liquids, Dowtherm A, Glycerol and Tri-o-cresyl-phosphate. Three conclusions were drawn from this study : Torpidity is a condition which applies to most liquids in the state of semi-purity irrespective of molecular size but it effects some liquids more than others. Torpidity is caused by small quantities of contaminants, the nature of which is not yet known and may be highly specific. Finally, torpidity is conspicuous when the surface is rapidly depleted of vapour.

Sommaire : L'auteur étudie l'effet de souillure de la surface d'un liquide en évaporation et introduit la conception du caractère torpide d'une telle surface.

Article by K. C. D. Hickman *Industr. Engng. Chem.* **44**, Aug. 1952 1892-1902

Studies in High Vacuum Evaporation. Comparison of High Vacuum Stills and Tensimeters

209/III

United States. Assuming that one can obtain the best separation of constituents of a mixture in a unit act of distillation, these values represent one theoretical plate. The authors assume that such one-plate data has been established for projective distillation using the 2-ethyl-hexyl-phthalate and 2-ethyl-hexyl-sebacate mixture as described in two previous articles (see Abstract Nos. 206/III, 207/III). In order to obtain equilibrium data of the same mixture, the authors endeavoured to develop a vacuum equilibrium still which facilitated the production of the highest reproducible values of relative volatility. These values would then represent one equilibrium plate. First a splashless equilibrium still, the barrel tensimeter, was designed, which consisted of a cylindrical container immersed in a thermostat and filled with the test liquid up to a quarter of its volume. Stirring was carried out by magnetic means controlled from outside. A tortuous neck restricting the escape of vapour connected to a cold finger condenser chamber. The flow of vapours through the neck could be interrupted. The values for relative volatility obtained with this still, stirring it slowly and restricting the escape of the vapour to one part in five hundred, were consistently higher than those reported previously, and it was assumed therefore that they represented one equilibrium plate. Operating the barrel tensimeter in the low micron range the experiments become very protracted and tedious. As it had been found previously (see Abstract No. 207/III) that equilibrant and projective distillation gave identical values below 2 μ Hg it was considered feasible to revert to projective distillation for work in the pressure region below 1 μ Hg. This led to the construction of the ' push-pull ' tensimeter. This still consisted of two projective evaporators processing the same liquid simultaneously from an expanding and contracting surface. The design originated from observations laid down in a previous paper (see Abstract No. 208/III). A full description and diagram of the apparatus are given. It was found that the relative volatilities from the expanding surface were higher than any so far recorded, including those obtained from the barrel tensimeter. On the other hand, relative volatilities from the contracted surface were smaller. The difference between the two rose with temperature and rate of evaporation. The fact that stretching the surface laterally can raise the separatory power above the presumed limit may lead to deliberate surface constructions which effect favoured separation of constituents. As a result of the experiments a curve could be drawn connecting vapour composition with temperature for the system EHP-EHS over an extended range. This curve represented one molecular plate, and, over a limited range, one equilibrium plate. This datum line was used for the assessment of other stills, such as centrifugal, falling-film and pot stills. The laboratory 5-inch centrifugal molecular still followed the datum line up to very high rates of distillation (40 g./sec./sq.m.) as long as the liquid was stirred. At very low rates the relative volatilities fell below the datum line. The falling-film still examined proved satisfactory over a certain range but fell off beyond 4 g. The unstirred and unjacketted pot still fell far below, registering $\frac{1}{3}$ to $\frac{1}{2}$ plate.

Sommaire : Tenant compte des conclusions présentées dans les articles (voir résumés Nos. 206/III et 207/III) les auteurs continuent par la description des distillateurs spécialement construits, dont la performance est définie pour représenter une plaque d'équilibre et faciliter une évaluation critique de la performance d'autres formes de distillateurs.

Article by K. C. D. Hickman & D. J. Trevoy *Industr. Engng. Chem.* **44**, Aug. 1952, 1903-1911

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

High Vacuum Multi-Stage Distillation Method and Apparatus

210/III

United States. The apparatus described is intended for molecular distillation but not restricted to it. It is claimed to represent an improvement of the design described in U.S. Patent No. 2,234,166, where reflux is obtained by gravity flow from a central and stationary condensing surface on to an outer and spinning frustro-conical evaporation surface. In the new design the central condensing surface rotates and the outer, annular evaporating surface is stationary. The distilland is fed on to the heated surface which is made up of a series of frustro-conical sections representing various stages. The residue of the distilland at any one stage flows by gravity to the next one below. The vapours travel horizontally in an inward direction across an annular highly evacuated space and condense on the cooled rotating surface formed as an inverted frustrum of a cone diverging upward. Here, due to the rotation of the condenser the liquid, forming a film, is subjected to a centrifugal force. This force is countered by a centripetal force. As a result the liquid is driven upwards along the condenser surface until it reaches the top of the respective distillation stage. From there it is thrown on to the stationary evaporating surface of the next stage. The distillate is withdrawn at the top and the residue at the bottom of the still. The material to be processed may be fed directly to the top, bottom or any intermediate stage of the still. This is governed by the composition of the feed material and the fractionation desired. The operational pressure should be 0.1 to 0.001 mm. Hg. The condenser is kept at a low temperature by liquid coolants circulated through the condenser at atmospheric pressure.

D. B. Luten, Jr. & Shell Development Co. *U.S. Pat.* 2,606,146

Sommaire : Construction d'un distillateur à étages multiples lequel par ses caractèristiques appartient à la catégorie des alambics à espace court non obstrué.

Directional Cold Finger Condenser

211/III

United States. A special design is described of a cold finger condenser of the type ordinarily used in small molecular pot stills. In order to restrict the action of the condenser to those molecules which come directly from a certain area of the distilland, the bottom of the finger is concave in shape and fitted with a circular retaining lip projecting inward. The finger is filled with oil, and a resistance coil is immersed in it. After starting the pump the coil is energised to heat the oil to a temperature well above that of the distilland. When stability has been reached in operation, the oil is exchanged with a coolant and as a result condensation takes place. However, all molecules not ' seen ' by the bottom face of the finger will condense on the side walls of the finger and will drop back into the pot whereas the ' selected ' condensate will be retained by the lip. At the end of the distillation process, before the vacuum is broken, the coolant is removed and the finger warmed up to near room temperature in order to prevent condensation of water from the ambient air. This introduces slight inaccuracies in the measurements but in the case of long runs these are immaterial and do not affect the value of the apparatus as a precision instrument suitable for high vacuum tensimetry.

Note by D. J. Trevoy *Analyt. Chem.* **24**, Aug. 1952 1382-1383

Sommaire : Description de la construction d'un condenseur du type intérieur qui facilite les mesures précises des performances de distillation dans une région choisie sur la surface du distillant dans un alambic à vide.

Versatile Electronic Manostat

212/III

United States. In connection with work chiefly concerned with the fractionation of paraffin mixtures a sensitive manostat was required to control the vaporisation rate in a Hyper-Cal column and Stedman-type column still. The operational pressures ranged from atmosphere to 30 mm. Hg. Salt water manostats were not suitable because the vapour pressure of the solution was too high for vacuum operation. Mercury would not give the desired sensitivity due to its density and sulphuric acid was unsafe. Finally dry ethylene glycol was chosen with a few drops of sulphuric acid added to it in order to increase the electrical conductivity. This material had a low vapour pressure, a low density, and little tendency to absorb paraffin vapours. A U-tube was used for the manostat. Electrical contact was made with platinum wire electrodes. One was sealed into the still wall below the liquid level, and the other was suspended from a fine gold plated brass chain and touched the surface of the liquid. The upper end of the chain was held by platinum wire sealed into a windlass made from the inner member of a 12 mm. standard tapered joint. One arm of the U-tube was joined to the still and the other to the manometer and the vacuum system. A change of the vaporisation rate made or broke the electrical contacts in the manostat which, in turn, caused a relay to operate and to alter the current supply to the heater of the still. The manostat described gave satisfactory service for more than 2,500 hours but its operational value under high vacuum conditions has not been explored.

Note by W. E. Erner & K. N. Campbell *Analyt. Chem.* **24**, July 1952 1232-1233

Sommaire : Construction d'un manostat sensible lequel peut être utilisé à la pression athmosphèrique ou sous vide de quelques mm. Hg développé spécialement en vue de contrôler la vitesse d'évaporation de mélanges paraffinés dans une colonne de distillation.

Shaft Seal for a Vacuum Apparatus

213/III

See Abstract No. : 149/II

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III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

39 — MISCELLANEOUS PROCESSES — 39

Production of Ammonium Nitrate by Continuous Vacuum Crystallisation — 214/III

United States. The operation of a continuous vacuum crystallisation plant is described for the production of fertiliser-grade ammonium nitrate crystals. The process is based on a method described previously by Miller and Saeman, and is safer and more economical than the graining process. The sequence of operations is given as follows: Neutralisation of nitrate acid with ammonia, purification and concentration of the ammonium nitrate solution, crystallisation, drying and bagging. The purified ammonium nitrate solution is concentrated to 79% content of ammonium nitrate in forced-circulation vacuum evaporators operating at a pressure of 2.4 inch. Hg with a capacity of 7,000 lb. of water vapour per hour, four units working in parallel. Crystallisation is carried out in five vacuum units, each designed to produce 105 tons of ammonium nitrate crystals per day. They consist essentially of a suspension container, a centrifugal circulation pump, a vaporiser, and a barometric leg. The container holds 23,000 gallons of the suspension. The vaporiser is 9 ft. in diameter. The feeding rate is 50 gallons per minute. The suspension is circulated from the container to the vaporiser where it is supersaturated by evaporative cooling. The supersaturated solution is returned to the bottom of the suspension container to prevent the crystals from settling out. Excess crystal nuclei collecting on the surface are removed, and the suspension is recirculated to the vaporiser. The finished crystalline products are collected continuously by air lifts from the suspension container. 65% of the final crystals average + 20 and 97% + 35 mesh in size. Operation can be continuous for several months without de-salting. The crystallisers operate in the temperature range of 90.1°F to 100°F in order to produce form–III crystals instead of form–IV which would be produced below 90.1°F. The product consists of single, rounded crystals containing a minimum of 33% nitrogen.

Article by W. C. Saeman, I. W. McCamy & E. C. Houston *Industr. Engng. Chem.* **44**, Aug. 1952 1912-1915

Sommaire: Détails d'une installation de productions récemment érigée, pour la manufacture de cristaux de nitrate d'ammonium selon une méthode de cristallisation continue sous vide, basée sur les principes de production proposés par Miller et Saeman.

A Laboratory-Scale Continuous Flash Evaporator — 215/III

See Abstract No.: 201/III

Frozen Concentrated Pineapple Juice — 216/III

Hawaii. A new plant worth one million dollars for the production of frozen concentrate of pineapple juice has been put into operation. The processing in the plant is somewhat different from that of orange juice processing, for instance, with regard to the method of recovering volatile esters and re-introducing them into the final product. Another special feature of the plant is the facilities it offers for 24-hours continuous operation, although fresh juice for processing is received during 6 hours of the day only. The process consists of seven steps as follows: (1) Fresh juice is prepared in the usual manner. (2) Inactivation of enzymes by heat treatment and pumping to the concentration plant. (3) A small amount is collected in a separate tank for blending at a later stage. The greater part is fed into a high vacuum low temperature triple-effect evaporator for partial concentration and separation of the volatile esters. (4) The esters are treated in a special vacuum fractionating still. The concentrated esters are stored at a temperature of 35°F. (5) The partially concentrated juice is returned to the evaporator for a second pass at a slower rate resulting in a 66° Brix concentration. During this operation pressure in the first effect averages 50 mm. Hg, in the second 35 mm. Hg, and in the third 25 mm. Hg. (6) Blending. This is effected by mixing set quantities of single strength cut-back juice and volatile flavour concentrate to the 66° Brix juice concentrate reducing the density of the final product to 47° Brix. (7) Canning and freezing. The latter process is carried out in an air blast tunnel 86 ft. long, 16 ft. wide, and fitted with a 10 ft. wide conveyor. The tunnel operates at –40°C for 18 hours per day. The remaining 6 hours it is used for freezing pineapple chunks.

Article by A. A. Smyser *Food Engng.* **24**, Aug. 1952 72-169, 170, 172

Sommaire: Description détaillée d'une installation de production à grande échelle, de jus d'ananas, congelé concentré.

Study of Vacuum Fumigation Techniques — 217/III

See Abstract No.: 197/III

Method and Apparatus for Fusing Dielectric Material in a Partial Vacuum by High-Frequency Electric Fields — 218/III

See Abstract No.: 196/I

On New Methods of Insulating Very Low Temperature Apparatus — 219/III

See Abstract No.: 202/I

The Analysis of Heat Transfer through Thermal-Insulating Materials — 220/III

See Abstract No.: 201/I

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40 — GASES AND VAPOURS — 40

A High Vacuum Flow Analogy 68/IV
See Abstract No. : 181/I

A Summary of the Present Status of Flow and Heat Transfer in Rarefied Gases 69/IV
See Abstract No. : 178/I

Drag on a Rotating Cylinder at Low Pressures 70/IV
See Abstract No. : 179/I

Nozzle Characteristics in High Vacuum Flow—Rarefied Gas Dynamics 71/IV
See Abstract No. : 180/I

Free Molecular Flow in the Sample Inlet to the Mass Spectrometer 72/IV

United States. Honig has established that there is a state of free molecular flow in the ion source of the mass spectrometer but this state is governed by the conditions in the sample reservoir and in the leak. The author, basing his article on Honig's work, studies the requirements in the latter parts of the inlet system. Molecular flow obtains if the flow rate in the leak is proportional to the partial pressure of each gas in the reservoir, and is established, according to Knudsen, if the diameter of the orifice is no more than 1/10 of the length of the mean free path of the gas at the higher pressure. The quantity of gas going through a molecular leak each second is $Q = 2.86d^2p(T/M)^{\frac{1}{2}}$ where d is the diameter of the orifice in cm., p the pressure of the gas in the reservoir, T the absolute temperature, and M the molecular weight. Figures are given for CO_2. The maximum desirable rate of flow Q, through the leak of the particular instrument used was assessed at 0.01 µl./sec. The mean free path of the gas, L_0, is determined by $1/L_0 = \sqrt{2}\pi n\delta^2$ where n is the number of molecules per cm.^{3} and δ the molecular diameter. The latter equals 4.6Å for CO_2. At 1 micron Hg pressure and 25°C. $L_0 = 3.34$ cm. and at any other pressure $L = 3.34/p$. Having specified the rate of flow to be 0.01 μl./sec. and having stipulated that the diameter of the orifice should be no more than 1/10 of the mean free path, the above formula can be used to give the orifice dimension required for the maximum pressure allowable in the case considered, *i.e.* d = 0.00405 cm., at p = 82.5 μ Hg. The author reports on experiments verifying molecular flow through a leak using hydrogen. A Norton leak was employed, projecting to the centre of the sample reservoir and connected to the mass spectrometer by a length of 40 cm. of 0.8 cm. diameter tubing. Plotting the effusion of the gas from the reservoir and the peak heights observed against time shows that the slopes of both curves were constant over the entire pressure range, *i.e.*, the leak rate was constant. Since the pressure ratio of p_t (reservoir) to p_h (peak height) was constant, the ion current was directly proportional to the gas pressure in the sample reservoir over the entire pressure range. Plotting the logarithms of the half times of gases of various molecular weights against the logarithms of the molecular weights a straight line was obtained, indicating that the rate of effusion through the leak was inversely proportional to the square root of the molecular weight. The three conditions for the existence of molecular flow were fulfilled. Finally, the author discusses flow in a mass spectrometer leak detector and investigates the effects of the rate of decay of pressure in the sample reservoir.

Sommaire : Etude théorique et expérimentale de l'écoulement libre moléculaire dans l'entrée d'un spectromètre de masse.

Article by P. D. Zemany *J. Appl. Phys.* **23**, Aug. 1952 924-927

Measurement of the Thermal Accommodation Coefficients of Gases 73/IV
See Abstract No. : 213/I

The Threshold Gas Pressure Required to Sustain a Stable Arc in a Magnetic Field 74/IV
See Abstract No. : 195/I

Unusual Vacuum Flasks 75/IV
See Abstract No. : 159/II

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41 — FREEZE DRYING — 41

Experiments with an Apparatus for Lyophile Desiccation of Micro-Organisms 76/IV

Holland. The author describes a freeze-drying apparatus of special design and observations made when using it for the freeze drying of micro-organisms. All experiments were carried out with a strain of *E. coli*. In order to ascertain how the temperature as such affects the rate of survival, various methods of freezing and thawing were applied and investigated in a special series of experiments. The test samples were all suspended in 0.25 ml. skimmed milk and all frozen material was dried in the same way. The dry material was suspended in 25 ml. of broth. The values shown in the two tables reproduced below apply to 1/20 ml. of this suspension. The number of surviving bacteria was counted by means of roll tube cultures prepared in accordance with the method of Julius. Rapid freezing was carried out by immersing the bottle with the test fluid in ethyl-glycol at a temperature of –35°C. During the process the bottle was revolved rapidly around its own axis resulting in a thin layer of frozen material which formed on the wall of the container within a few seconds. Rapid thawing was obtained by placing the ampoules in a water bath at 37°C. The results of the experiments are given in Tables I and II.

Table I
The Number of Bacteria Surviving Various Methods of Freezing

Treatment	*Surviving Bacteria*				*Average*
Original Culture (control)	2017	1007	1980	1972	1994
Slowly frozen to –10°C	42	41	39	38	40
Rapidly frozen to –10°C	337	317	315	310	320
Slowly frozen to –35°C	278	278	276	272	276
Rapidly frozen to –35°C	1881	1843	1843	1824	1848

Table II
The Number of Bacteria Surviving Various Methods of Thawing

Treatment	*Surviving Bacteria*				*Average*
Original Culture (control)	2017	1007	1980	1972	1994
Thawed slowly	327	324	323	320	323
Thawed rapidly	936	918	918	900	913

A further table is added showing the effects of rapid and repeated temperature changes. The following conclusions have been arrived at: Freezing should be rapid. The freezing temperature should be –35°C or lower. This temperature should be maintained for the whole of the desiccation period, or if this is not possible, desiccation should be rapid. No deterioration could be discovered if the frozen material was kept at that temperature for some time. The freeze drying apparatus used in these experiments was evacuated by a mechanical pump which provided an ultimate pressure of 10^{-3} to 10^{-2}mm. Hg. The moisture was collected in a liquid air trap. The pressure was measured with a McLeod manometer. Two kinds of manifold were used, one made from glass and one made from metal. The apparatus in question was designed to facilitate sterilisation by steam at 120°C. This is considered preferable to sterilisation by alcohol, as many micro-organisms survive treatment with alcohol, and may cause cross infection. In order to find the optimum drying period a series of experiments was carried out with drying times ranging from 15 mins. to 3 hours but the results were not conclusive.

Article by H. L. Wolff *Anton Leeuwenhoek*, 18, 1952 113-124

Sommaire : L'auteur rend compte de ses observations, faites lors de la lyophilisation d'une colonie de Colibacilles E dans un appareil spécial.

Homotransplantation of Tracheal Segments Preserved by Lyophilisation. An Experimental Study 77/IV

See Abstract No. : 219/I

A New Method for Getting and Preserving Organ Powders Destined for Therapeutics by Using Low Temperature, Desiccation and Vacuum 78/IV

France. The organs are frozen soon after being taken. They are then pulverised at low temperature (between –112° and –148°F) in refrigerated grinders. The powder thus obtained is vacuum-dried at –7.6°F ; the process is completed with the addition of silica gel grains which are subsequently sifted. Preservation in air-tight bottles in inert conditions.

(*Bull. Inst. Int. du Froid*)

Article by H. S. Tival *Le Genie Civil* March 1951 96

Sommaire : Méthode nouvelle pour la préservation d'organes pulvérisés employant basse température et vide.

Cantilever Microtome 79/IV

See Abstract No. : 157/II

IV — Special Subsidiary Subjects — IV
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Abstract No. and References

45 — ELECTRONICS — 45

Ion Producing Mechanism 80/IV

See Abstract No. : 225/I

The Oak Ridge 86-Inch. Cyclotron 81/IV

United States. Details are given of the 86-inch high current proton cyclotron of the fixed frequency type which was completed at the end of 1950 at Oak Ridge. The cyclotron produces an internal beam in excess of 1 mA of protons at 24 MeV. The machine has a pole face diameter of 86 inch. a maximum beam radius of 33 inch, a magnetic field of 9,000 oersteds, a resonance frequency of 13.4 megacycles per sec., a peak dee-to-dee potential of 300 kV to 500 kV, a dee-to-dee gap of 4 inch. and a dee width of 8 inch. The orientation of the magnetic field is different from other machines, being at right angles. The dees are suspended vertically. The U-shaped magnetic yoke is 40 × 62 inch. in cross section, and weighs 250 tons. The vacuum system consists of three 20-inch oil diffusion pumps each equipped with an 8-inch booster pump and backed by a 15 h.p. mechanical pump. The total pumping speed of the system is 15,000 litres/sec. at a pressure of 0.03 micron Hg. The oscillator is of the grounded-grid self-excited type using a single Federal 134 tube rated at 400 kW and 150 kW plate dissipation. In order to control ' ion loading ' a dee-bias system with a negative 1,000 V potential on the dees is employed. Radioactivity of the dees is reduced by applying a layer of graphite. As a result the dees can be approached quite safely a few hours after operation. Two types of targets used in the cyclotron are mentioned : in the case of the first the beam passes through an 0.015 inch. aluminium window and into the watercooled target directly behind the window. This is a satisfactory arrangement for metallic targets where only a few microampere-hours are required. For larger irradiations the target is made as large as possible and placed at an angle of 2-5° to the incident beam. Aluminium targets 6 × 10 inch. have been used in beams of 30 kW or more. Good cooling is indispensable. The induced activities are high, *e.g.* a zinc target bombarded for one hour at 400 μA gave 50 R/hr at one meter distance.

Sommaire : Détails de la construction du cyclotron bâti à Oak Ridge et mis en service a la fin de 1950.

Article by R. S. Livingston *Nature* **170**, 9.8.52 221-223

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Dissociation of Molecular Hydrogen Ions in the Cyclotron 82/IV

See Abstract No. : 210/I

Failure of Vacuum Insulation at Low Field Strengths 83/IV

United Kingdom. Pre-breakdown currents in vacuum insulated systems are usually believed to be due to field emission. The authors, however, noted that if the electrodes in continuously pumped systems are not heat-treated, a form of conduction occurs at unexpectedly low field strengths which cannot be due to field emission. The current depends critically on anode conditions and the voltage required is only slightly changing if the gap length is varied. The mechanism of the new phenomenon is still unexplored but resembles the ' electron loading ' experienced in accelerating tubes in the megavolt field.

Sommaire : Des phénomènes d'effondrement du pré-vide observés par l'auteur sont discutés.

Letter by D. S. Clifford, R. L. Fortescue, J. Roberts *Nature* **170**, 20.9.52 503

The Effect of Surface Treatment on Electric Breakdown Between Copper Electrodes at 50 Cycles at Very Low Pressures 84/IV

See Abstract No. : 194/I

47 — GLASS PROCESSING — 47

Metal Coatings on Glass or Quartz not Intended for Mirrors or Decoration 85/IV

Germany. Several methods are discussed of coating quartz glass with a metal so that soldering operations can be carried out on or near evacuated components, *e.g.* thermionic valves. A number of recent German patents is quoted. One method described in the German patent No. 680371 provides for the metal to be applied in the form of a paste and fired on to the quartz glass by heating it dielectrically for a short period to a temperature which is higher than the softening temperature of the glass but lower than the melting temperature of the metal. A vacuum furnace is used. Heating is applied for about one minute. As the quartz glass heats up slowly the metal is only for some 10 seconds above the softening temperature of the glass. Cooling should be rapid. Metals suitable for this method are tungsten, molybdenum and tantalum. Where heavy gauge leads have to be connected to thin wires sealed into a glass body it is often impracticable to use screws or soft soldering. The method described in German patent No. 721088 facilitates hard soldering and provides for a shield made from a material of high thermal conductivity which is placed around the area where soldering is to be carried out. The shield protects the glass surface from exposure to excessive heat but in special cases it may be advisable to cool the glass with an air blast during operation.

Sommaire : Différentes méthodes allemandes pour la métallisation de verre ou quartz qui comprennent la soudure sur des appareils à haut vide.

Article by Dr. Borchard *Glas Email Keramo Tech.* **2**, 15.3.51 68

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A.E.C. Reports	U.S. Atomic Energy Commission Reports,
A.E.C.D. Reports	Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
A.E.R.E. Reports	U.K. Atomic Energy Research Establishment Reports, Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1, England.
Amer. J. Phys.	American Journal of Physics, American Institute of Physics, Michigan State College, East Lansing, Michigan, U.S.A.
Amer. Sci.	American Scientist, Society of the Sigma Xi and Scientific Research Society of America, 54, Hillhouse Avenue, New Haven, Conn., U.S.A.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 1155, Sixteenth Street, N.W., Washington 6, D.C., U.S.A.
Analyt. Chim. Acta	Analytica Chimica Acta, Elsevier Publishing Co., Amsterdam, Holland.
Anton. Leeuwenhoek	Antonie van Leeuwenhoek, Nederlandsch Tijdschrift voor Hygiëne, Microbiologie en Serologie, N.V. Swets en Zeitlinger, Keizersgracht 471, Amsterdam C., Holland.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Arch. Tech. Messen	Archiv für Technisches Messen, R. Oldenbourg Verlag, Lotzbeckstrasse 2 b, München, Germany.
Bell. Lab. Rec.	Bell Laboratories Record, Bell Telephone Laboratories Inc., 463, West Street, New York 14, N.Y., U.S.A.
Brit. J. Appl. Phys.	British Journal of Applied Physics, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
B.S.I.R.A. Bulletin	Bulletin of the British Scientific Instrument Research Association, British Scientific Instrument Research Association, Information Department, 'Sira,' Southill, Elmstead Woods, Chislehurst, Kent, England.
Bull. Classe Sci.	Bulletin de la Classe des Sciences, Academie Royale de Belgique, Palais des Academies, 1, Rue Ducale, Bruxelles, Belgium.
Bull. Inst. Int. du Froid	Bulletin de l'Institut International du Froid, Institut International du Froid, 177, Boulevard Malesherbes, Paris, 17e, France.
Bull. Soc. Roy. Belge Ing. Industr.	Bulletin de la Société Royale Belge des Ingénieurs et des Industriels, 3, Rue Ravenstein, Bruxelles, Belgium.
Bur. Mines Reports	Bureau of Mines Reports, U.S. Dept. of the Interior, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
Canad. Chem. Process.	Canadian Chemical Processing, Westman Publications Ltd., 137, Wellington Street, West Toronto, Ontario, Canada.
Canad. J. Phys.	Canadian Journal of Physics, The National Research Council, Ottawa, Canada.
Canad. J. Technol.	Canadian Journal of Technology, The National Research Council, Ottawa, Canada.
Chemical Abstracts	Chemical Abstracts, American Chemical Society, c/o Ohio State University, Columbus 10, Ohio, U.S.A.
Chem. Age	Chemical Age, Benn Bros., Bouverie House, 154, Fleet Street, London, E.C.4, England.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1., England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Verlag Chemie G.m.b.H. Weinheim/Bergstrasse, Hauptstrasse 129, Germany.
Chem. Process Engng.	Chemical & Processing Engineering, Leonard Hill, Ltd., 17, Stratford Place, London, W.1, England.
Chem. Weekbl.	Chemisch Weekblad, N.V.D.V. Centens Uitgevirs-Maatschappij, Sarthatikade 12, Amsterdam C., Holland.
Colloque Int. Spectrograph., Strasbourg	Colloque International de Spectrographie de Strasbourg tenu à l'Université de Strasbourg, Groupement pour l'avancement des méthodes spectrographiques des produits métallurgiques, 1, Place Saint-Thomas-d'Aquin, Paris 7e, France.
C. R. Acad. Sci., Paris	Comptes Rendus des Séances de l'Academie des Sciences, Messrs. Gauthier-Villar, 55, Quai des Grands-Augustine, Paris, France.

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Dokl, Akad. Nauk, S.S.S.R.	Doklady Akademii Nauk Soyuza Sovetskikh Sotsialisticheskikh Respublik, Izdatel'stvo Akademii Nauk, S.S.S.R., Moscow, Russia.
D.S.I.R. Reports	Department of Scientific Industrial Research Reports, Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1, England.
Elect. Engng.	Electrical Engineering, American Institute of Electrical Engineers. 500, Fifth Avenue, New York 36, N.Y., U.S.A.
Electronics	Electronics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Elektroteknikeren	Elektroteknikeren, Elektroteknisk Tidskrift for Lys og Kraft, Telefoni og Telegrafi, Solvgade 10, Copenhagen K., Denmark.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Engng. J.	Engineering Journal, The Engineering Institute of Canada, 2050, Mansfield Street, Montreal, Canada.
Erdöl u. Kohle	Erdöl und Kohle, Industrieverlag Hernhausen Komm. Ges., Alsterufer 4/5, Hamburg 36, Germany.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Food Technol.	Food Technology, Institute of Food Technologists, 119-123, West Park Avenue, Champaign, Ill., U.S.A.
Gen. Elect. Rev.	General Electrical Review, General Electric Co., 1, River Road, Schenectady 5, N.Y., U.S.A.
Le Génie Civil	Le Génie Civil, Revue Techniques Générale des Industries Francaises et Etrangères, 5, rue Jules-Lefebvre, Paris 9e, France.
Glas Email Keramo Tech.	Glas Email Keramo Technik, P.O. Box Hamburg Rahlstedt 1, Germany.
Glas-und Hochvakuum Tech.	Glas-und Hochvakuum Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Indian J. Phys.	Indian Journal of Physics and Proceedings of the Indian Assoc. for the Cultivation of Science, Indian Association for the Cultivation of Science, Jadavpur, Calcutta 32, India.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 1155, Sixteenth Street, N.W., Washington 6, D.C., U.S.A.
Instruments	Instruments, Instruments Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
Invest. Phys.	Investigationes Physicae, 40, Carlton Crescent, Summer Hill, Sydney, N.S.W., Australia.
J. Acoust. Soc. Amer.	Journal of the Acoustical Society of America, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Amer. Ceramic Soc.	Journal of the American Ceramic Society, American Ceramic Society, 2525, N. High Street, Columbus 2, Ohio, U.S.A.
J. Amer. Oil Chem. Soc.	Journal of the American Oil Chemists Society, The American Oil Chemists' Society, 35, E. Wacker Drive, Chicago 1, Illinois, U.S.A.
J. Appl. Chem.	Journal of Applied Chemistry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Brit. I.R.E.	Journal of the British Institution of Radio Engineers, British Institution of Radio Engineers, 9, Bedford Square, London, W.C.1, England.
J. Chem. Educ.	Journal of Chemical Education, Div. of Chemical Education of the American Chemical Society, Scripps Institution, La Jolla, California, U.S.A.
J. Chem. Phys.	Journal of Chemical Physics, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Chem. Soc.	Journal of the Chemical Society, The Chemical Society, Burlington House, Piccadilly, London, W.1, England.
J. Chim. Phys.	Journal de Chimie Physique et de Physico-Chimie Biologique, Société de Chimie Physique, 10, Rue Vauquelin, Paris (5e), France.

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J. Comp. Path. & Therapeutics	Journal of Comparative Pathology and Therapeutics, University of Liverpool, Liverpool 3, England.
J. Dairy Sci.	Journal of Dairy Science, American Dairy Science Association, N. Queen Street and McGovern Avenue, Lancaster, Pa., U.S.A.
J. Electrochem. Soc.	Journal of the Electrochemical Society, The Electrochemical Society, 235, West 102nd Street, New York 25, N.Y., U.S.A.
J. Electrodeposit. Tech. Soc.	Journal of the Electrodepositors Technical Society, Institute of Metal Finishing, 27, Islington High Street, London, N.1, England.
J. Franklin Inst.	Journal of the Franklin Institute, The Franklin Institute of Pennsylvania, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Hygiene	Journal of Hygiene, 200, Euston Road, London, N.W.1, England.
J. Inst. Metals	Journal of the Institute of Metals, The Institute of Metals (Inc.), 4, Grosvenor Gardens, London, S.W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers, Inc., 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Phys. Radium	Le Journal de Physique et le Radium, La Société Francaise de Physique, 10, Rue Vauquelin, Paris (5e), France.
J. Phys. Soc., Japan	Journal of the Physical Society of Japan, Physical Society of Japan, c/o Dept. Physics, Faculty of Science, Tokyo University, Bunkyo-ku, Tokyo, Japan.
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
J. Sci. Instrum.	Journal Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
J. Sci. Res. Inst., Japan	Journal of the Scientific Research Institute, Scientific Research Institute Ltd., 31, Kamifujimae-cho, Komagome, Bunkyo-ku, Japan.
J. S.M.P.T.E.	Journal of the Society of Motion Picture and Television Engineers, Society of Motion Picture and Television Engineers, 40, West 40th Street, New York 18, N.Y., U.S.A.
J. Thoracic Surg., St. Louis	Journal of Thoracic Surgery, C. V. Mosby Co., 3207, Washington Boulevard, St. Louis 3, Mo., U.S.A.
Lab. Practice	Laboratory Practice, United Trade Press Ltd., 9, Gough Square, Fleet Street, London, E.C.4, England.
Leather & Shoes	Leather and Shoes, The Rumpf Publishing Co., 300, West Adams Street, Chicago 6, Illinois, U.S.A.
Mach. Design	Machine Design, The Penton Publishing Co., Penton Buildings, Cleveland 13, Ohio, U.S.A.
Machinery	Machinery, Machinery Publishing Co., Clifton House, 83-117, Euston Road, London, N.W.1, England.
Malay. Pharm. J.	Malayan Pharmaceutical Journal, Malayan Pharmaceutical Association, Post Box 664, Singapore.
McGraw-Hill Digest	McGraw-Hill Digest, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Finish.	Metal Finishing, Finishing Publications Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Prog.	Metal Progress, American Society of Metals, 7301, Euclid Avenue, Cleveland, 3, Ohio, U.S.A.
Metal Treatment	Metal Treatment, Industrial Newspapers Ltd., 49, Wellington Street, Strand, London, W.C.2, England.
Metalloberfläche	Metalloberfläche, Carl Hanser Zeitschriftenverlag, G.m.b.H., München 27, Germany.
Metallurgia	Metallurgia, Kennedy Press Ltd., 31, King Street West, Manchester 3, England.
Métaux Corrosion Industr.	Métaux Corrosion Industries, Editions Métaux, 32, Rue du Maréchal-Joffre, Saint-Germain-en-Laye, (S.-et-O.), France.
Mfg. Chem.	Manufacturing Chemist, Leonard Hill Ltd., Stratford House, 9, Eden Street. London, N.W.1, England.

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Nature	Nature, Macmillan & Co. Ltd., St. Martin's Street, London, W.C.2, England.
N.B.S. Tech. News Bull.	National Bureau of Standards Technical News Bulletin, Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
Netherlands Milk Dairy J.	Netherlands Milk Dairy Journal, N.V.D.V. Centens Uitgevirs-Maatschappij, Sarthatikade 12, Amsterdam C., Holland.
N.M.R.I. Reports	Naval Medical Research Institute, Naval Medical Centre, Bicester, Maryland, U.S.A.
Nuclear Science Abstr.	Nuclear Science Abstracts, U.S. Atomic Energy Commission, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
Nucleonics	Nucleonics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
O.N.R. Reports	Office of Naval Research Reports, Office of Naval Research, Department of the Navy, Washington, D.C., U.S.A.
Optik	Optik, Wissenschaftliche Verlagsgesellschaft m.b.H., Stuttgart, Germany.
Overseas Engr.	Overseas Engineer, Temple Press Ltd., 27, Shaftesbury Avenue, London, W.1, England.
P.D.A. Journal	P.D.A. Journal, Photographic Dealers Association, P.D.A. House, 46, Bloomsbury Street, London, W.C.1, England.
Philips Tech. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, England.
Physica	Physica, Martinus Nijhoff, The Hague, Holland.
Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Proc. Gen. Discuss. on Heat Transfer	Proceedings of the General Discussion on Heat Transfer, Institution of Mechanical Engineers, Storey's Gate, St. James's Park, London, S.W.1, England.
Proc. Inst. Electr. Engrs.	Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, Strand, London, W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Royal Society, The Royal Society, Burlington House, Piccadilly, London, W.1, England.
Prod. Finish.	Product Finishing, Sawell Publication Ltd., 4, Ludgate Circus, London, E.C.4, England.
Rech. Aéron.	La Recherche Aéronautique, Office Nationale d'Etudes et de Recherches Aéronautiques, 25, Avenue de la Division Leclerc, Chatillon/Bagneux (Seine), France.
Refrig. Coldstor. Air Conditng.	Refrigeration, Coldstorage and Air Conditioning, 12, Sprod Avenue, Toorak Gardens, Adelaide, South Australia.
Research	Research, Butterworths Scientific Publications, 4-6, Bell Yard, Temple Bar, London, W.C.2, England.
Rev. Alumin.	Revue de l'Aluminium, 77, Boulevard Malesherbes, Paris (8e), France.
Rev. Opt.	Revue d'Optique, 3-5, Boulevard Pasteur, Paris (15e), France.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
Schiffbau Tech.	Schiffbau Technik, Verlag Technik, Unter den Linden 12, Berlin, N.W.7, Germany.
Science	Science, American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W., Washington 5, D.C., U.S.A.
Science Abstracts	Science Abstracts, Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
Seifen, Ole Fette Wachse	Seifen, Ole Fette Wachse, Augsburg 2, Postfach, Germany.
Steel	Steel, Penton Publishing Co., Penton Building, Cleveland 13, Ohio, U.S.A.
Steelways	Steelways, American Iron & Steel Institute, 350, Fifth Avenue, New York 1, N.Y., U.S.A.

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Abbreviations and Publishers of Journals Quoted in this Volume

Contd.

Tekn. Tidskr.	Teknisk Tidskrift, P.O.B. 841, Stockholm 1, Sweden.
The Times (Industr.Suppl.)	The Times Industrial Supplement, The Times Publishing Co., Printing House Square, London, E.C.4, England.
The Times (Science Suppl.)	The Times Science Supplement, The Times Publishing Co., Printing House Square, London, E.C.4, England.
Trans. A.I.E.E.	Transactions of the American Institute of Electrical Engineers, 33, West 39th Street, New York, 18, N.Y., U.S.A.
Trans. A.S.M.E.	Transactions A.S.M.E., American Society of Mechanical Engineers, 29, West 39th Street, New York 18, N.Y., U.S.A.
Trans. Faraday Soc.	Transactions of the Faraday Society, The Faraday Society, Aberdeen University Press Ltd., 6, Upper Kirkgate, Aberdeen, Scotland.
Univ. Calif. Tech. Rep.	University of California Technical Reports, Institute of Engineering Research, University of California, Berkeley, Calif., U.S.A.
Le Vide	Le Vide, Société Francaise des Ingénieurs des Techniciens du Vide, 44, Rue de Rennes, Paris (6e.), France.
World Refrig.	World Refrigeration, 181, Kensington High Street, London, W.8, England.
World's Paper Trade Rev.	World's Paper Trade Review, Stonehill and Gillis Ltd., 329, High Holborn, London, W.C.1, England.
Z. Angew. Phys.	Zeitschrift für Angewandte Physik, Springer Verlag/Berlin-Charlottenburg Jebenstrasse 1, Germany.
Z. Electrochem.	Zeitschrift für Electrochemie, Verlag Chemie G.m.b.H., Weinheim/Bergstrasse, Haupstrasse 129, Germany.
Z. Naturforsch.	Zeitschrift für Naturforschung, Verlag der Z. für Naturforschung, Mathildenstrasse 29, Tübingen, Germany.
Z. Phys.	Zeitschrift für Physik, Springer Verlag/Berlin-Charlottenburg Jebenstrasse 1, Germany.
Z. V.D.I.	Zeitschrift des Vereines Deutscher Ingenieure, Prinz Georg Strasse 77/79, Düsseldorf, Germany.
Z. Wiss. Photograph. Photophys. Photochem.	Zeitschrift für Wissenschaftliche Photographie, Photophysik und Photochemie, Johann Ambrosius Barth Verlag, Leipzig, Germany.

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